

NMR Probes Installation

Varian Liquids, Solids, Microimaging NMR Probes

Pub. No. 01-999034-00, Rev. B0798

varian  YEARS
nuclear magnetic resonance instruments

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Applicability of manual:

Probes used with ^{UNITY}*INOVA*, *MERCURY* VxWorks-Powered (shortened to *MERCURY-VX* throughout this manual), *MERCURY*, *UNITYplus*, *GEMINI 2000*, *UNITY*, *VXR*, and Gemini NMR spectrometer systems

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F0597 – Added AutoSwitchable and new Nano probes

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SAFETY PRECAUTIONS

The following warning and caution notices illustrate the style used in Varian manuals for safety precaution notices and explain when each type is used:

WARNING: *Warnings are used when failure to observe instructions or precautions could result in injury or death to humans or animals, or significant property damage.*

CAUTION: *Cautions are used when failure to observe instructions could result in serious damage to equipment or loss of data.*

Warning Notices

Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these warnings, or with specific warnings elsewhere in Varian manuals, violates safety standards of design, manufacture, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

WARNING: **Persons with implanted or attached medical devices such as pacemakers and prosthetic parts must remain outside the 5-gauss perimeter of the magnet.**

The superconducting magnet system generates strong magnetic fields that can affect operation of some cardiac pacemakers or harm implanted or attached devices such as prosthetic parts and metal blood vessel clips and clamps.

Pacemaker wearers should consult the user manual provided by the pacemaker manufacturer or contact the pacemaker manufacturer to determine the effect on a specific pacemaker. Pacemaker wearers should also always notify their physician and discuss the health risks of being in proximity to magnetic fields. Wearers of metal prosthetics and implants should contact their physician to determine if a danger exists.

A table on the back of the *5-Gauss Warning Sign* shows the typical 5-gauss stray field for various magnet systems.

WARNING: **Keep metal objects outside the 10-gauss perimeter of the magnet.**

The strong magnetic field surrounding the magnet attracts objects containing steel, iron, or other ferromagnetic materials, which includes most ordinary tools, electronic equipment, compressed gas cylinders, steel chairs, and steel carts. Unless restrained, such objects can suddenly fly towards the magnet, causing possible personal injury and extensive damage to the probe, dewar, and superconducting solenoid. The greater the mass of the object, the more the magnet attracts the object.

Only nonferromagnetic materials—plastics, aluminum, wood, nonmagnetic stainless steel, etc.—should be used in the area around the magnet. If an object is stuck to the magnet surface and cannot easily be removed by hand, contact Varian service for assistance.

A table on the back of the *10-Gauss Warning Sign* shows the typical 10-gauss stray field for various magnet systems.

Warning Notices (*continued*)

WARNING: Only qualified maintenance personnel shall remove equipment covers or make internal adjustments.

Dangerous high voltages that can kill or injure exist inside the instrument. Before working inside a cabinet, turn off the main system power switch located on the back of the console.

WARNING: Do not substitute parts or modify the instrument.

Any unauthorized modification could injure personnel or damage equipment and potentially terminate the warranty agreements and/or service contract. Written authorization approved by a Varian Associates, Inc. product manager is required to implement any changes to the hardware of a Varian NMR spectrometer. Maintain safety features by referring system service to a Varian service office.

WARNING: Do not operate in the presence of flammable gases or fumes.

Operation with flammable gases or fumes present creates the risk of injury or death from toxic fumes, explosion, or fire.

WARNING: Leave area immediately in the event of a magnet quench.

If the magnet dewar should quench (sudden appearance of gasses from the top of the dewar), leave the area immediately. Sudden release of helium or nitrogen gases can rapidly displace oxygen in an enclosed space creating a possibility of asphyxiation. Do not return until the oxygen level returns to normal.

WARNING: Avoid helium or nitrogen contact with any part of the body.

In contact with the body, helium and nitrogen can cause an injury similar to a burn. Never place your head over the helium and nitrogen exit tubes on top of the magnet. If helium or nitrogen contacts the body, seek immediate medical attention, especially if the skin is blistered or the eyes are affected.

WARNING: Do not look down the upper barrel.

Unless the probe is removed from the magnet, never look down the upper barrel. You could be injured by the sample tube as it ejects pneumatically from the probe.

WARNING: Do not exceed the boiling or freezing point of a sample during variable temperature experiments.

A sample tube subjected to a change in temperature can build up excessive pressure, which can break the sample tube glass and cause injury by flying glass and toxic materials. To avoid this hazard, establish the freezing and boiling point of a sample before doing a variable temperature experiment.

Warning Notices (*continued*)

WARNING: Support the magnet and prevent it from tipping over.

The magnet dewar has a high center of gravity and could tip over in an earthquake or after being struck by a large object, injuring personnel and causing sudden, dangerous release of nitrogen and helium gasses from the dewar. Therefore, the magnet must be supported by at least one of two methods: with ropes suspended from the ceiling or with the antivibration legs bolted to the floor. Refer to the *Installation Planning Manual* for details.

WARNING: Do not remove the relief valves on the vent tubes.

The relief valves prevent air from entering the nitrogen and helium vent tubes. Air that enters the magnet contains moisture that can freeze, causing blockage of the vent tubes and possibly extensive damage to the magnet. It could also cause a sudden dangerous release of nitrogen and helium gases from the dewar. Except when transferring nitrogen or helium, be certain that the relief valves are secured on the vent tubes.

WARNING: On magnets with removable quench tubes, keep the tubes in place except during helium servicing.

On Varian 200- and 300-MHz 54-mm magnets only, the dewar includes removable helium vent tubes. If the magnet dewar should quench (sudden appearance of gases from the top of the dewar) and the vent tubes are not in place, the helium gas would be partially vented sideways, possibly injuring the skin and eyes of personnel beside the magnet. During helium servicing, when the tubes must be removed, follow carefully the instructions and safety precautions given in the manual *Varian Magnet Installation and Maintenance*.

Caution Notices

Observe the following precautions during installation, operation, maintenance, and repair of the instrument. Failure to comply with these cautions, or with specific cautions elsewhere in Varian manuals, violates safety standards of design, manufacture, and intended use of the instrument. Varian assumes no liability for customer failure to comply with these precautions.

CAUTION: Keep magnetic media, ATM and credit cards, and watches outside the 5-gauss perimeter of the magnet.

The strong magnetic field surrounding a superconducting magnet can erase magnetic media such as floppy disks and tapes. The field can also damage the strip of magnetic media found on credit cards, automatic teller machine (ATM) cards, and similar plastic cards. Many wrist and pocket watches are also susceptible to damage from intense magnetism.

A table on the back of the *5-Gauss Warning Sign* shows the typical 5-gauss stray field for various magnet systems.

Caution Notices (*continued*)

CAUTION: Check helium and nitrogen gas flowmeters daily.

Record the readings to establish the operating level. The readings will vary somewhat because of changes in barometric pressure from weather fronts. If the readings for either gas should change abruptly, contact qualified maintenance personnel. Failure to correct the cause of abnormal readings could result in extensive equipment damage.

CAUTION: Never operate solids high-power amplifiers with liquids probes.

On systems with solids high-power amplifiers, never operate the amplifiers with a liquids probe. The high power available from these amplifiers will destroy liquids probes. Use the appropriate high-power probe with the high-power amplifier.

CAUTION: Take electrostatic discharge (ESD) precautions to avoid damage to sensitive electronic components.

Wear grounded antistatic wristband or equivalent before touching any parts inside the doors and covers of the spectrometer system. Also, take ESD precautions when working near the exposed cable connectors on the back of the console.

Radio-Frequency Emission Regulations

The covers on the instrument form a barrier to radio-frequency (rf) energy. Removing any of the covers or modifying the instrument may lead to increased susceptibility to rf interference within the instrument and may increase the rf energy transmitted by the instrument in violation of regulations covering rf emissions. It is the operator's responsibility to maintain the instrument in a condition that does not violate rf emission requirements.

Chapter 1. Probe-Related Hardware

Sections in this chapter:

- 1.1 “Overview of NMR Probe Hardware” [this page](#)
- 1.2 “Installing the Pneumatic System” [page 17](#)
- 1.3 “Installing the Probe and Upper Barrel” [page 18](#)
- 1.4 “Pneumatics/Tachometer Box” [page 20](#)

This chapter introduces probe hardware and describes how to install related hardware, such as the pneumatic system, upper barrel, and Pneumatics/Tachometer box.

1.1 Overview of NMR Probe Hardware

The basic probe system consists of three parts (see [Figure 1](#)):

- The *upper barrel* is used as a mechanism to deliver the sample into the center of the magnet. This part also contains the apparatus for spinning the sample. The upper barrel is inserted into the magnet from the top until it seats on the room temperature (RT) shim coil. Typically, it remains in the magnet.
- The *probe*, the lower part of the probe system, is inserted into the magnet from below and secured with thumbscrews. The top of the probe mates to the upper barrel. The proper alignment of these two parts, the upper barrel and the probe, is important for proper spinning and probe performance.
- The *room temperature shim coils* surround the probe and are used to adjust magnet field homogeneity.

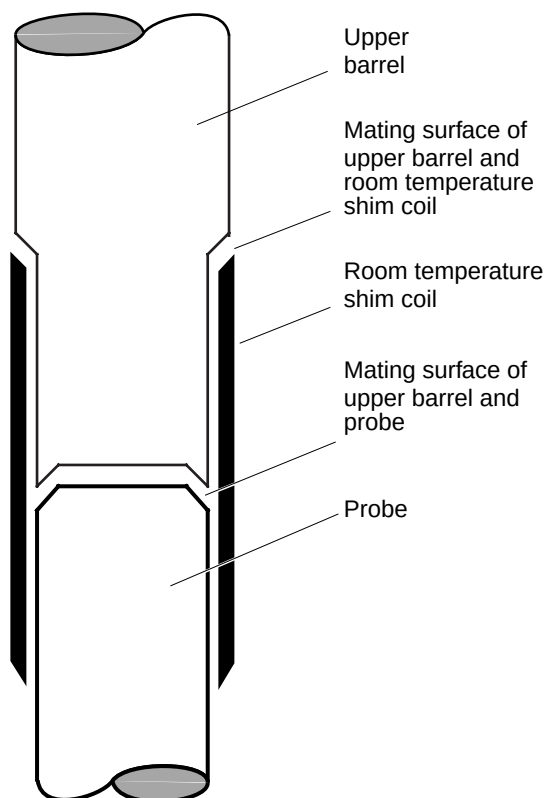


Figure 1. Probe, Upper Barrel, and RT Shim Coil Configuration

The procedures for connecting and tuning each probe are basically similar. The connectors referred to in the cable connection instructions are found on the left magnet leg (or magnet-console interface) and, if a ^{UNITY}INOVA, ^{UNITYplus} system, or a ^{UNITY} system with an Oxford magnet, on the preamplifier assembly bolted to the magnet stand. **Figure 2** shows examples of various preamplifier configurations and **Figure 18** shows the assembly for ^{UNITY}INOVA and ^{UNITYplus} systems.

Each probe in this manual is identified by the *probe part number* (the number shown on the identification label attached to the probe) and the *probe order number* (the part number used for ordering the probe). The probe order number identifies a *probe kit*, which includes the probe assembly, a sample kit, and the probe installation manual.

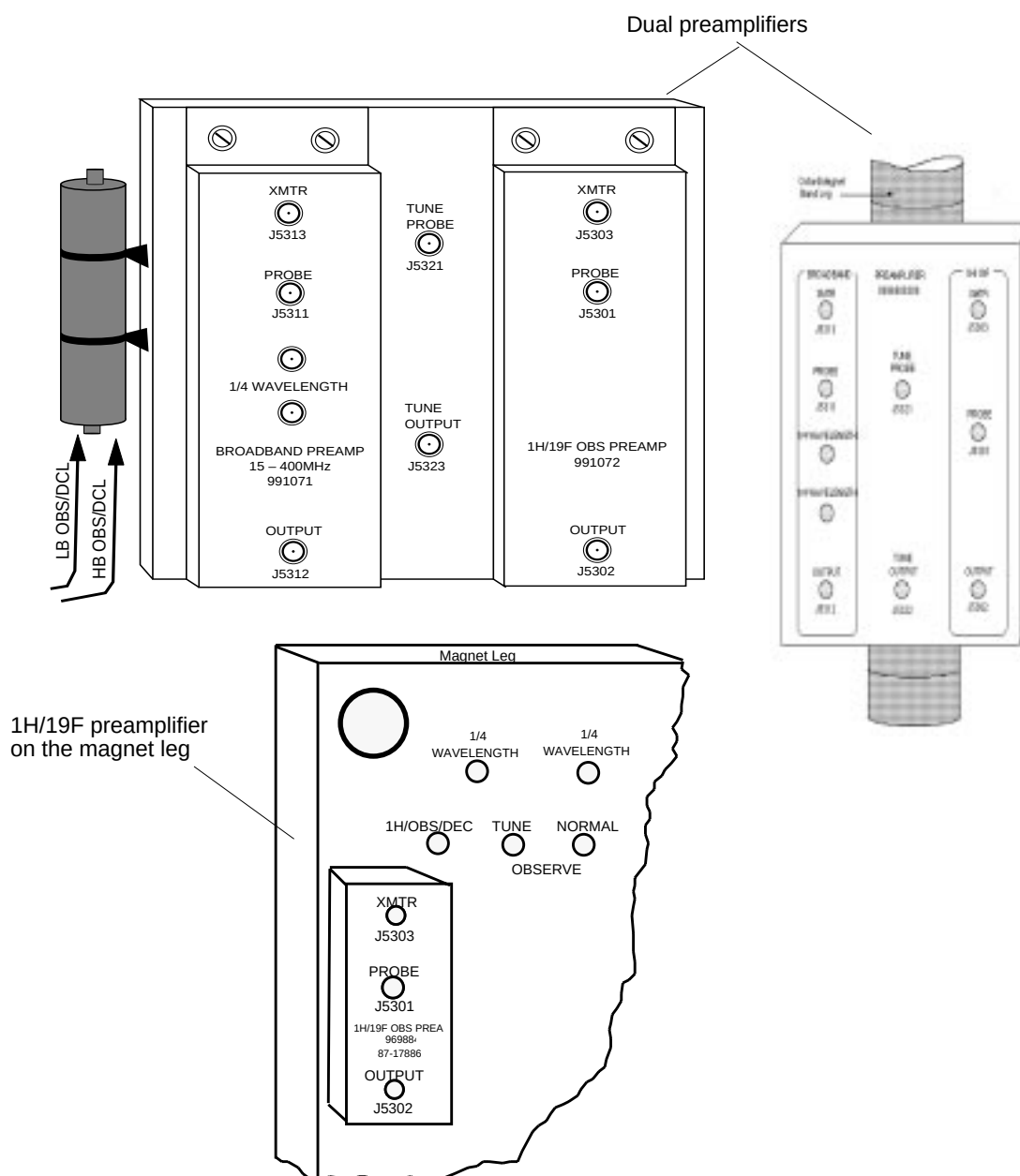


Figure 2. Dual and ¹H/¹⁹F Preamplifiers

1.2 Installing the Pneumatic System

The pneumatic system for Varian and Oxford magnets uses quick-disconnect manifold, valves, and push-in fittings. The fittings enable quick connects and disconnects by holding the insert with two fingers and pushing the tubing in or pulling it out. Magnets are shipped with a pressure regulator and oil filter for the air supply as well as a pressure regulator and oil filter for the nitrogen supply.

The following procedure assumes that the probe is installed. If the probe is not yet installed, skip step 1 until the probe is installed (see [Section 1.3, “Installing the Probe and Upper Barrel,”](#) on page 18).

WARNING: Do not make any connections when the pneumatic system is under pressure.

1. Connect the COOLING Tygon tube to the probe (see [Figure 3](#)). Use the cooling air meter, located inside the right-hand leg of the Varian magnet and on the preamplifier housing of the Oxford magnet, to maintain a flow rate of about 10 liters/minute.

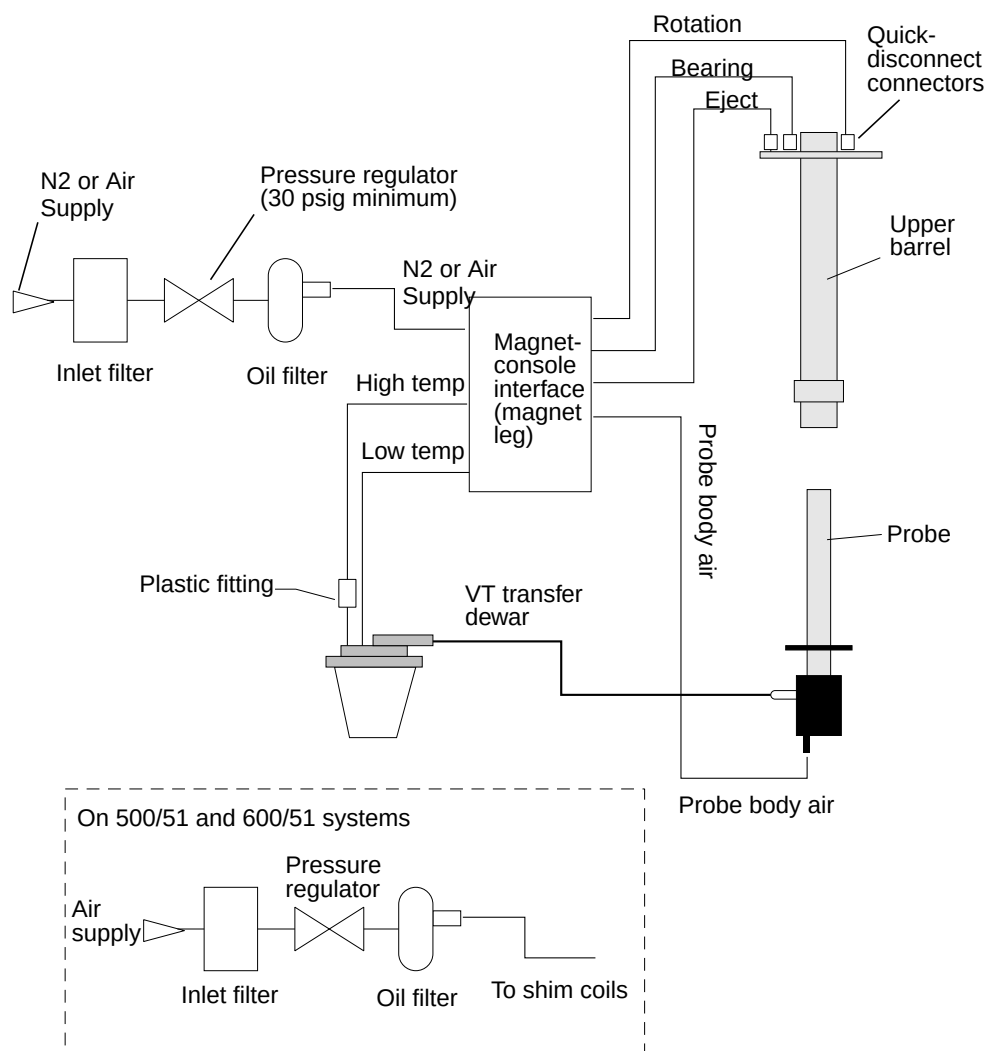


Figure 3. Pneumatic System Connection Diagram

2. Connect the ROTATION, EJECT, and BEARING tubing to the upper barrel assembly with quick-disconnect fittings. These fittings have an internal check valve so air flow shuts off when they are disconnected.
3. Connect the MANUAL EJECT inlet tubing to the left-leg manual eject valve, and connect the MANUAL EJECT outlet tubing to the manual eject valve outlet (see Figure 4).
4. Install a 1/4 inch push-in type fitting to each oil filter outlet.
5. Connect pressure regulators with filters (air only) to both the air and nitrogen gas sources.
6. Connect the supply tubing and nitrogen supply tubing together, then connect the tubing to either the air or nitrogen pressure regulator and filter system.

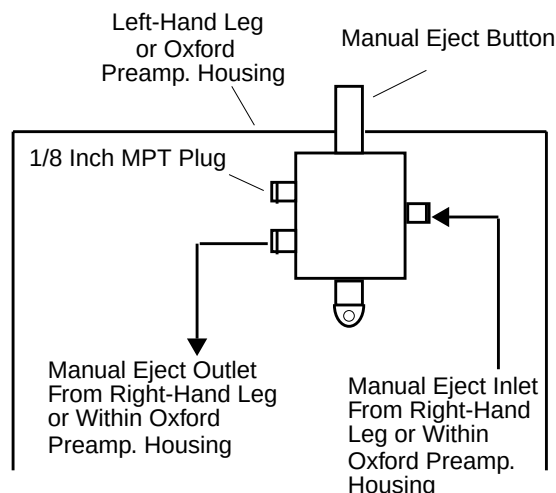


Figure 4. Manual Eject Valve

CAUTION: The VT transfer dewar is made of glass, is not flexible, and should be handled with care.

7. For variable temperature (VT) operation, connect the “high temperature” tubing to the heat exchanger (VT bucket) outlet, and connect the transfer dewar from the heat exchanger outlet to probe inlet. The plastic fittings supplied with the VT kit allows a solid connection between “low temperature” plastic tubing and the heat exchanger (VT bucket) inlet.
 8. Increase the air or nitrogen gas pressure. If you find a leak between a tube and a fitting, refer to Chapter 7, “Maintenance and Troubleshooting,” for troubleshooting procedures..
- Do not mix the two gases (air and nitrogen) within the probe. It is best to use one gas for the whole system.

1.3 Installing the Probe and Upper Barrel

Use the procedure outlined in this section to install the upper barrel and probe for any probe described in this manual. Refer to the section that describes the probe for further details on connections and tuning.

WARNING: Keep your head away from the helium and nitrogen exit tubes and the probe sample outlet. Do not look down the upper barrel unless the probe is removed from the magnet.

CAUTION: Probes contain fragile ceramic and glass parts. Avoid shock or pressure that might damage these components. Do not drop the probe. Do not drop anything down the top of the probe barrel.

To install the probe and upper barrel:

1. Determine that a test sample spins in the upper barrel before inserting the upper barrel in the magnet:
 - a. Connect the ROTATION, EJECT, and BEARING tubing to the upper barrel assembly and start the air flow. (Refer to the Section 1.2, "Installing the Pneumatic System," on page 17.)
 - b. With the top of the upper barrel tilted to no more than 30° from horizontal and one hand placed over the bottom, place the sample and rotor into the top.
 - c. Slowly raise the upper barrel to vertical. The sample should spin freely.
 - d. Cover the top of the upper barrel with your hand and carefully tip the upper barrel to eject the sample.
2. Carefully insert the upper barrel in the magnet without the probe in place.
Take care not to pinch off any of the air hose lines. The upper barrel makes contact with the shim coil set at point B in [Figure 1](#). Do *not* tighten the thumbscrews on the upper barrel collar at this time.
3. With a nonmagnetic hex-key wrench, loosen the three setscrews in the probe mounting flange until the flange can be moved but the weight of the probe does not cause the probe to slide out of the flange.
4. Insert the probe into the magnet until it makes contact with the upper barrel at point A in [Figure 1](#). Push the probe and upper barrel assembly up and tighten the mounting flange thumbscrews. Be sure to tighten the thumbscrews equally.
If the probe mounting plate has not been adjusted yet for the magnet, adjust the plate as follows:
 - a. Loosen the three setscrews in the mounting flange with the nonmagnetic hex-key wrench supplied, and move the mounting plate to 1/4 inch (6 mm) from the lower end.
 - b. Insert the probe into the magnet until it meets a stop. Do not force the probe upward, because the upper barrel, held in place by only an O-ring, will move.
 - c. Rotate the probe until the rf connectors are positioned, and then rotate the mounting plate.
 - d. Push the probe upwards until the plate is flat against the magnet surface.
 - e. Secure the two flange thumbscrews to the magnet and gently tighten (1 to 2 inch-pound torque) the three setscrews on the flange.
5. Rotate the probe until the VT connecting dewar (positioned on one of the magnet legs) is in line with the lower probe dewar extension. Push down on the upper barrel until contact is made at point B once more.
6. Attach the upper barrel to the upper magnet flange by tightening the thumbscrews on the upper barrel collar, then tighten the setscrews equally on the probe mounting flange. Be sure not to over-tighten the setscrews.

CAUTION: The setscrews should be just tight enough so the probe is firmly held in the flange. Be careful to not over-tighten because it is possible to dent the shield.

1.4 Pneumatics/Tachometer Box

The room temperature (RT) and variable temperature (VT) Pneumatics/Tachometer Boxes are used with Varian RT CP/MAS, Varian VT CP/MAS, and Doty Scientific solids probes as well as with Varian Nano•nmr probes. The Pneumatics/Tachometer Box handles all air/gas supply distribution to the probe. The supply line is permanently connected to the wall supply, which must be clean, dry air. The wall supply should be at a pressure not exceeding 120 psig (8 bar) and be filtered to 0.6 micron.

CAUTION: Failure to maintain a clean and dry air supply shortens probe life.

This section contains general information about the RT and VT Pneumatics/Tachometer Boxes and installation instructions.

RT CP/MAS Pneumatics/Tachometer Box

Figure 5 shows the Pneumatics/Tachometer Box for RT (room temperature) CP/MAS probes. The box is mounted on a magnet leg. The two hoses coming out of the left side of the box are connected to the probe ROTATION/DRIVE and BEARING connectors. These connectors are of high-pressure, quick-disconnect types.

The RT Pneumatics/Tachometer Box comes in two versions: either with or without an independent power supply. If the box does not have its own power supply (for CP/MAS operation), the tachometer cable (P6001) is disconnected from the upper barrel and connected to the Molex socket on the box o provide power to the electronics in the box. A tachometer sensor in the probe is also connected to the box with the cable provided.

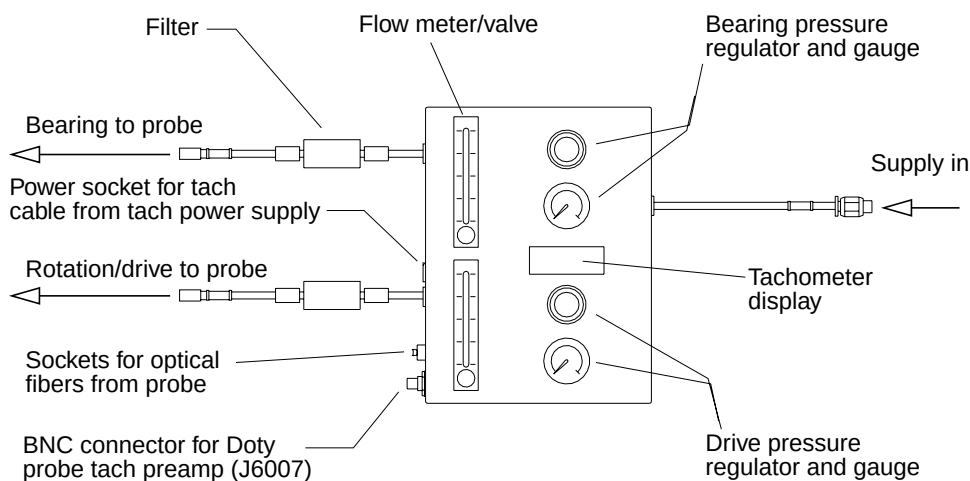


Figure 5. Pneumatics/Tachometer Box for RT CP/MAS Probes

VT CP/MAS Pneumatics/Tachometer Box

Figure 6 shows a Pneumatics/Tachometer Box for VT (variable temperature) CP/MAS probes. This box is mounted on a leg of the magnet in a convenient position. The four hoses coming out of the left side of the pneumatics box are connected to the probe ROTATION/

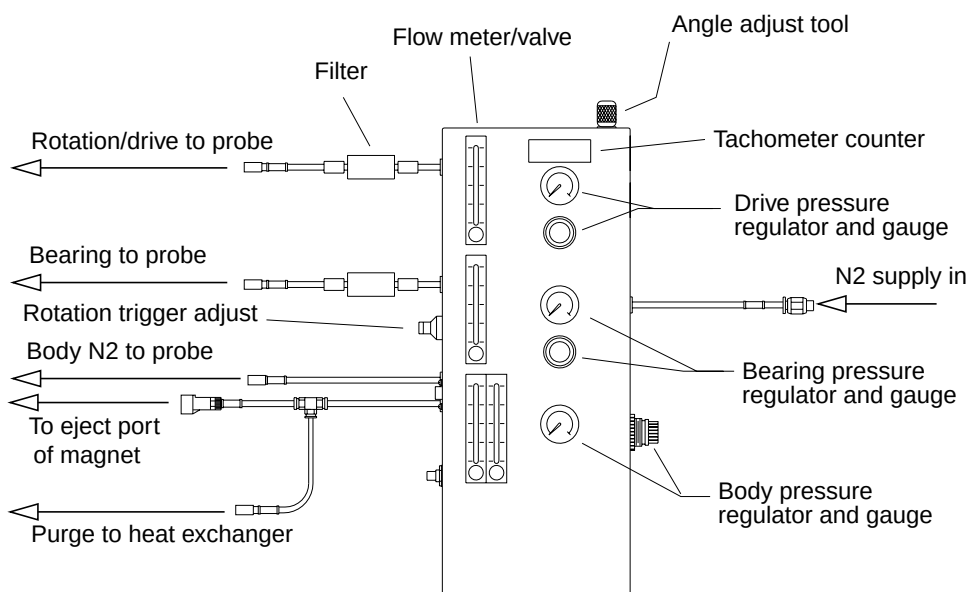


Figure 6. Pneumatics/Tachometer Box for VT and RT CP/MAS Probes

DRIVE, BEARING, BODY GAS, and the EJECT port of the magnet. These connectors are of a high-pressure, quick-disconnect type.

Installing the Pneumatics/Tachometer Box

To install an RT or VT Pneumatics/Tachometer box:

1. Using the hardware provided in the Pneumatics/Tachometer Box kit, install the box on the magnet leg.

CAUTION: Use only dry N₂ for any VT work.

2. Connect the inlet hose of the Pneumatics/Tachometer Box to a clean, dry supply of compressed air or nitrogen. This supply should be at 90 psig continuous pressure.
3. Close both the rotation/drive and bearing pressure regulators, and turn on the supply. Check for air leaks by slowly opening both pressure regulators until a pressure of 10 psig shows on the gauges. No air should escape through the hoses. If air escapes, check for grit in the probe connectors. When this test is complete, close both regulators.
4. Open the check valve of the air connector on each hose from the Pneumatics/Tachometer Box to the probe. This is done by inserting a thin tool (such as a small flat-head screwdriver) into the connector and pressing down until a click is felt.
5. Turn the bearing regulator to show 10 psig. There should be a good flow rate shown on the flow meter. If the flow is low, turn the needle valve on the flow meter counter-clockwise until a good flow is obtained. Repeat this procedure for the drive supply.

Note that under normal circumstances, the bearing and drive flowmeter needle valves are left in the full open position and are not used for control.

WARNING: When operating under high pressure, make sure the valves on older flow meters are not too loose. Older flow meters have removable flow knobs that can fly off if loosened too much when under high pressure.

6. Install the Pneumatics/Tachometer Box power supply in the back of the rf cabinet.
7. Install the cable from the power supply to the Pneumatics/Tachometer Box, routing it with the other cables running to the magnet. Feed the coax terminated with a SMB connector through the cabinet to the front of the digital card cage.
8. Plug the SMB connector into the Acquisition Controller board at the EXT TIME BASE position.
9. Plug the probe fiber optic cables into the Pneumatics/Tachometer Box in one of the following configurations:
 - *Varian probes* – Switch to VA optic.
 - *Doty probes* – On the Pneumatics/Tachometer Box, set the 3-position switch to EXT NEG.
10. Plug the supply cord from the Pneumatics/Tachometer Box power supply into the 110 Vac distribution panel inside the rf cabinet (on the rear center).
11. Make sure that power is supplied to the Pneumatics/Tachometer Box by checking for light from the optical connector of the Pneumatics/Tachometer Box. The LCD display on the Pneumatics/Tachometer Box should also display a decimal point.
12. For Doty probes only, install the dummy optical plug (if supplied) into the optical receiver connector of the Pneumatics/Tachometer Box.
13. For Doty probes only, connect the Doty tachometer amplifier optical output position to the Pneumatics/Tachometer Box external input. Plug the power supply into a source of 110 Vac. Move the switch on the tachometer amplifier marked optical off to the position away from its label.
14. Restart the data station and the acquisition console.
15. Use the `config` program to set Rotor Sync to Present as described in the *VNMR and Solaris Software Installation Manual*.
16. Connect a CP/MAS probe to the Pneumatics/Tachometer Box air hoses.
17. For Varian VT CP/MAS probes, connect the body N₂ to the probe and adjust to 20 LPM 5 LPM for VT operation. We recommend that about 5 LPM body N₂ be used for ambient operation.

Replacing the Air/Gas Filter

Replace the blue disposable 0.6 micron particle filter (Part No. 27-180354-00) after it has become discolored (turned red).

To install a new filter:

1. Disconnect the filter by pressing the movable sleeve on the gas hose fitting toward the tube fitting (you may need a small crescent wrench) and pulling the filter out of the tube fitting.
2. Insert a new filter, with the arrow on the filter pointing towards the probe.
3. Return the movable sleeve to its original position.

Chapter 2. **Adjustments and Tuning**

Sections in this chapter:

- 2.1 “Adjusting Spinning and Air Pressure” this page
- 2.2 “Using the qtune Graphical Probe Tuning Program” page 25
- 2.3 “Tuning Probes with UNITYINOVA or UNITYplus” page 33
- 2.4 “Tuning Probes with MERCURY-Series or GEMINI 2000 Systems” page 37
- 2.5 “Tuning Probes with UNITY or VXR” page 40.
- 2.6 “Tuning Probes with Gemini Systems” page 43.
- 2.7 “Setting the Lock Frequency on 600-MHz Systems” page 45.
- 2.8 “Changing Samples or Solvents” page 46.
- 2.9 “Testing a Probe” page 46.
- 2.10 “Using Probe Calibration Files” page 46.

Although the information in this chapter applies to all probes, tuning procedures differ between spectrometers and are described in separate sections.

2.1 Adjusting Spinning and Air Pressure

For best results, operate the spinner (upper barrel) under the conditions shown [Table 1](#) (the “Standard” configuration column is for systems without the sample changer installed).

Table 1. Spinning Conditions With and Without a Sample Changer

<i>Item</i>	<i>Standard</i>	<i>With Sample Changer</i>
Upper barrel Part No.	00-948003-xx	00-958341-xx
Maximum sample size	16 mm	5 mm
Minimum sample size	5 mm	5 mm
Turbine color	green	white
System air pressure	26–28 psig ¹	18–20 psi
Bearing air valve	open about 1/2 turn	open about 1/4 turn
Slow drop air valve	open about 2 turns	closed
Sample depth ²	69 mm	124 mm
Spin air pressure	not applicable	3–8 psig

1. PFG upper barrels require greater air pressure, typically 26–45 psi.

2. Maximum depth from the spinner bottom to the sample tube bottom.

To Adjust the Cooling Air

Adjust the COOLING AIR valve (located on the right magnet leg) for best performance. The probe cooling air line is fed by this valve.

To Adjust the Bearing Air

Adjust bearing air as follows:

1. Turn off bearing air valve.
2. Turn the valve on again slowly until the console SPIN light starts flashing. Enter the `lock` routine of `acqi` and make sure the spin speed is displayed on the screen.
3. Open the bearing valve 1/16 turn more.

To Adjust System Air Pressure

Adjust the system air within the limits specified in [Table 1](#) as necessary to optimize sample insertion, ejection, and spinning. The VT air (or dry N₂ gas) line must be connected to the probe at all times when the probe is in use.

VT air is provided to the probe by placing the VT connecting dewar in line with the probe dewar extension. Set the VT ball valve meter to read 5–10 LPM to start and reset as necessary to optimize sample insertion, ejection, and spinning. *Failure to attach the necessary air components will cause difficulty in sample spinning and ejection, sample cooling (decoupler heating), and possibly shimming.*

Always use either the *insert* (`i`) and *eject* (`e`) commands or `acqi` to insert and eject the sample, except on *MERCURY*, *GEMINI 2000*, or Gemini systems without Computer-controlled Insert/Eject Module installed.

2.2 Using the qtune Graphical Probe Tuning Program

This section describes how to use the graphical probe tuning program (shown in [Figure 7](#)) for interactive NMR probe tuning on *UNITYINOVA* and *UNITYplus* systems, and on *MERCURY-VX* systems equipped with the graphical tuning upgrade.

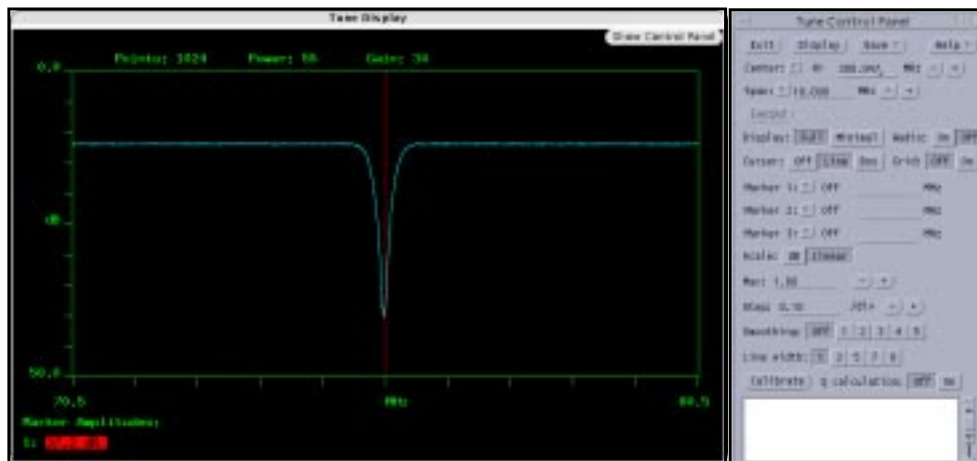


Figure 7. Tune Display Window and Tune Control Panel (qtune Program)

The *qtune* program runs on the host computer and provides the user with an interactive tuning method that provides separate information for matching and resonant frequency. This program is especially useful for tuning probes with complicated coil configurations, such as imaging probes.

After the system is put into tune mode, the reflected power from the probe passes through the directional coupler and is detected and digitized by the receiver circuitry: Any power that the receiver detects is reflected power. Taking one (or more) complex pair of data points at each frequency gives a data set that shows reflected power versus frequency. A coil tuned to a specific frequency (usually the frequency of the nucleus the user wants to observe) reflects little power at that frequency. The acquisition system then sweeps through the desired frequency range and gathers data on reflected power interactively. The user can adjust certain parameters in real-time during the experiment.

Tuning a Probe with qtune

This procedure describes how to use the *qtune* program to tune an NMR probe.

1. Set up the system for tuning:

UNITYINOVA and *UNITYplus* – leave the switch set to observe mode.

MERCURY-VX – connect the following cables:

- Connect the appropriate cable from the probe (J5102 or J5302) to the TUNE J5402 connector on the inside of the magnet leg.
- Connect the appropriate cable from the transmitter (J5602 or J5603) to the TUNE J5604 connector.
- Connect the appropriate cable from the receiver (J5303 or J5103) to the Q TUNE J5403 connector.
- Enter **tn= 'n' su**, where *n* is the nucleus to be tuned (e.g. tn= 'H1').

2. In the VNMR input window, enter **qtune(gain, power)**, where *gain* and *power* are appropriate gain and power values. Typical entries are **qtune(20, 65)** for *UNITYINOVA* and *UNITYplus*, and **qtune(0, 15)** for *MERCURY-VX*. Generally you want as low a gain value as possible with as high a power value as possible. If **qtune** is entered without arguments, the default values of 50 for *gain* and 60 for *power* are used.

The Tune Display and Tune Control windows open, similar to [Figure 7](#). The Tune Display is centered on the resonant frequency of the current experiment (*sfrq*).

To change the gain and power values, click Exit in the Tune Control Panel and reenter the **qtune** command with more appropriate values.

3. In the **Display** field, select **Full** or **Minimal**.
 - Full display shows the network-analyzer-like graph, as shown in [Figure 7](#).
 - Minimal display simplifies the tune display by showing numerical values instead of the graph, as shown in [Figure 8](#). When sweeping over a range of frequencies, the minimal display shows minimum reflected power and center frequency. When in CW mode, the minimal display shows the average reflected power and the current frequency. Several display controls on the Tune Control Panel are disabled in minimal display mode, including cursors, grid, and markers.
4. In the **Audio** field, select **On** or **Off**.

If set to On, a volume slider becomes available and a sound is generated that indicates how close the minimum reflection is to the center of the sweep window—the lower the pitch of the sound, the closer the minimum reflection is to the center. The sound immediately stops if the response lacks a discernible minimum reflection or if CW mode is set.

To use the audio capability requires:

 - An audio speaker on the Sun computer.

CW mode



Figure 8. Minimal Display Mode for the Tune Display

- The probe tuned well enough so that a clearly discernible minimum reflection exists in the signal.
 - A range of frequencies sent to the probe (i.e, audio requires sweep mode rather than CW mode).
5. Place a marker on the resonant frequency to which you want to tune the probe as follows (this option is not available in minimal display):

- a. In the Tune Control Panel (see [Figure 9](#)), click on the triangle next to one of the markers to open a pull-down menu ([Figure 13](#) shows the Marker 1 menu).

For more detail on using markers, see the “[Using Cursors, Grid, and Markers](#)” on page 29.

- b. In the menu, select the resonant frequency to which you are tuning the probe.

A marker corresponding to the selected frequency appears in the Tune Display window.

6. In the Tune Control Panel, type values as appropriate in the **Span**, **Scale**, and other fields on the Tune Control Panel.

Using the dB scale usually facilitates probe tuning. See the “[Adjusting the Span](#)” on page 28.

7. Adjust the tune and match capacitors while watching the Tune Display window. Use the match capacitor to increase the depth of the dip as much as possible. Use the tune capacitor to center the dip on the marker created in [step 5](#).

The dip displayed in the Tune Display window shows where little power is reflected at the frequency being observed. The depth of the peak shows the accuracy of the impedance matching of the probe coil to the transmitter and receiver. The horizontal location of the dip shows the frequency at which little power is reflected. The goal of probe tuning is to increase the depth of the peak (matching) while centering the dip at the desired frequency.

8. After the probe is tuned, click **Exit**. You are ready to begin the experiment.



Figure 9. Tune Control Panel

Adjusting the Center Frequency

The center frequency is the resonant frequency to which the probe is to be tuned. A list of center frequencies appears in the Center pull-down menu in the Tune Control Panel (see [Figure 10](#)). This list of frequencies is read from the VNMR nuctable file.

Note that any frequency you type into the field will not be read until you press Return. This applies to all text entry fields.

1. In the **Center** field, click on the triangle to open the pull-down menu, then select a frequency that equals or is close to the frequency you want.

- Adjust the frequency by typing a new value in the Center field or by clicking the – and + buttons.

The – or + buttons decrease or increase the value by the width of one span.

If the center frequency is either typed in or changed by the – or + button, user is displayed next to the frequency. If the typed value happens to correspond to a nuclear frequency in the pull down menu, that nucleus is displayed.

If the new center is too close to either of the system frequency limits, the span is decreased to allow the new center to be accepted, the message window will beep, and an error message will appear. If the specified center is past either frequency limit, the message window will beep, and an error message will appear.

Adjusting the Span

The span is the sweep width, in MHz, used in the Tune Display.

- To decrease or increase the span to the next in the series 1,2,5 10,20, etc., in the **Span** field, enter a value or click the – or + button.

Setting the span to less than 1000 Hz causes the message window to beep and an error message to appear.

Setting the span to more than maximum span causes the message window to beep and an error message to appear.

Setting the span to a value beyond the maximum or minimum frequencies causes the span to decrease, the message window to beep, and user warnings to appear.

- In the **Span** field, click on the triangle to open the Span pull-down menu (see [Figure 11](#)).
 - Select **Last Span** to return to the previous span value
 - Select **Maximum Span** to make the spectrometer sweep from the minimum to maximum allowable frequencies.
 - Select **CW** to temporarily stop frequency sweeping and to make the transmitter put out a CW signal. This sets the frequency to the currently selected center frequency. This mode is useful for checking the reflected power on the tune meter or for making other tests that require a fixed frequency.

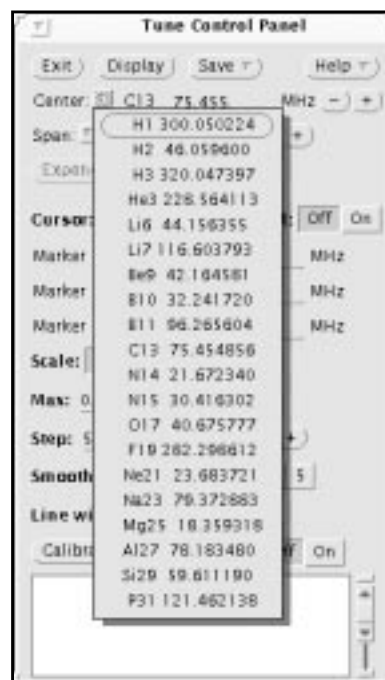


Figure 10. Pull-Down Menu For Center Frequencies



Figure 11. Pull-Down Menu For Span

Using Cursors, Grid, and Markers

Cursors and markers appear on the Tune Display window and are used the same way they are used in VNMR. Cursors and markers are color coded and the frequency positions of each are displayed on the bottom of the Tune Display. Cursor, grid, and marker controls are not available in minimal display.

Cursors

In the **Cursor** field, the Tune Control Panel provides three cursor modes (see Figure 12)



Figure 12. Cursor and Grid Controls

- Select **Off** to turn cursors off so that no cursors are displayed.
- Select **Line** to display a single cursor that specifies one frequency. The frequency position of the cursor and the signal amplitude at that frequency are displayed on the bottom of the Tune Display window.
- Select **Box** to display two cursors for use with the Expand button (for setting the span to a narrower range). After a region is expanded, the cursor mode switches back to the Off mode. The frequency positions of the cursors, as well as the delta and the signal amplitudes, are displayed on the bottom of the Tune Display window.

Grid

In the **Grid** field, select **Off** or **On** (see Figure 12) to control a grid display in the Tune Display window. The grid helps in reading the reflected power levels off of the graph. The grid does not slow down the drawing time of the graph.

Markers

In the **Marker 1**, **Marker 2**, and **Marker 3** fields, the Tune Control Panel provides three markers for marking fixed frequencies. Each marker has a pull-down menu (see Figure 13) that lists the same nuclear frequencies and the Center pull-down menu. Each marker also has an entry field for entering a frequency.

You can use markers for observing fixed frequencies, for example the two nuclear frequencies of a double tuned probe. You can then vary the span, and the markers will appear and disappear, depending on whether their frequencies are being scanned in the current experiment.

Attempting to set the markers to values beyond the system frequency limits causes the message window to beep and produces an error message.

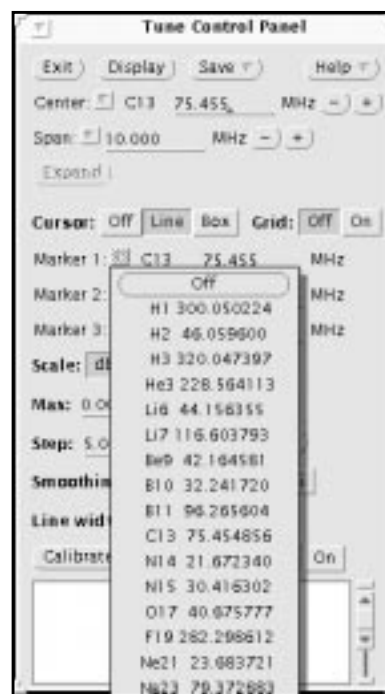


Figure 13. Pull-Down Menu for Marker 1

Changing the Vertical Scale

You can change the vertical scale of the Tune Display window by selecting whether the data is scaled in a linear or logarithmic manner (see Figure 14).

1. In the **Scale** field, the Tune Control panel provides two scaling modes:

- Select **dB** to provide a logarithmic scale in units of dB.
- Select **Linear** to a linear scale with arbitrary units.



Figure 14. Scale Controls

The dB scale shows a deeper dip for more accurate match adjustment.

2. In the **Max** field, type a value you want for the top line in the Tune Display.
Setting a Max value beyond system limits generates a beep and an error message.
3. In the **Step** field, type a value you want for the number of units per division mark of the current scale (dB or linear) on the y axis of the Tune Display.
Step helps by making the height of a probe resonance appear larger or smaller.
Setting a Scale value beyond system limits generates a beep and an error message.

Calculating the Q Value

Probe Q factor determines sensitivity. Q is defined as the frequency of the resonant circuit divided by the half-power bandwidth.

To calculate the Q value:

1. In the **Q calculation** field, select **On**.
The current Q value and resonant frequency appears at the top of the Tune Display window, and a horizontal cursor appears on the plot.
2. In the Tune Display window, use the middle mouse button to place the horizontal cursor on the base line (reflected power level outside of the resonance line).
The Q calculation appears at the top of the Tune Display window.

The Q value shown is determined as follows:

- The software finds the lowest point on the display and designates this as the resonance $V_{\min}$. The frequency displayed is, at best, only as accurate as the frequency difference between points. You must take this into account when quoting Q measurements.
- The software takes the level of the horizontal cursor as the baseline, or $V_{\max}$, which is assumed to be a frequency where all the rf energy is reflected by the probe.
- The frequencies ω_1 and ω_2 have the following signal level.

$$\frac{V_{\max}}{\sqrt{5}} \quad [\text{Eq. 1}]$$

- Q is calculated from the following equation, where ω_r is the resonant frequency

$$Q = \frac{\omega_r}{|\omega_1 - \omega_2|} \quad [\text{Eq. 2}]$$

- The software checks that the low point (the bottom of the dip) is at least 15 dB below the baseline. If this is not true, the calculated Q value is not accurate, and is, therefore, not reported (the string “- - -” appears in the Q value field). The resonance frequency, however, is still given.

Calibrating the Tune System

A Tune Calibration window is available for calibrating the tune system using a shorting load, an open load (no device attached), and a 50-ohm load.

To calibrate the tune system.

1. Set up the system for tuning:

UNITY *INOVA* UNITY*plus* – Leave the switch set to observe mode.

MERCURY-VX – Connect the following:

- Connect the appropriate cable from the probe (J5102 or J5302) to the TUNE J5402 connector on the inside of the magnet leg.
- Connect the appropriate cable from the transmitter (J5602 or J5603) to the TUNE J5604 connector.
- Connect the appropriate cable from the receiver (J5303 or J5103) to the Q TUNE J5403 connector.
- Enter **tn= 'n' su**, where *n* is the nucleus to be tuned (e.g., tn= 'H1 ').

2. Click the **Calibrate** button on the Tune Control Panel. The Tune Calibration window opens (see Figure 15).

You must run the calibration tests in the following order:

- Short Test
- Open Test
- 50 Ohm Test

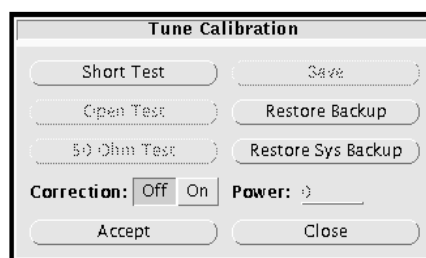


Figure 15. Tune Calibration Window

The test buttons are only available in this order.

The program stores only correction coefficients in files. Therefore, you will not be able to view old calibration sweeps after the experiments are finished.

3. In the **Correction** field, select **Off** or **On** to disable or enable calibration corrections, respectively. The default is enabled.

If valid calibration files are not present, this button is not available.

4. Connect a shorting load:
 - UNITY *INOVA* and UNITY*plus* – Connect a shorting load to the tune port.
 - MERCURY-VX – Connect a shorting load to TUNE J5402 on the inside (probe side) of the magnet leg.
5. Click the **Short Test** button.
6. Remove the shorting device from the tune port.
7. With no load on the tune port, click the **Open Test** button.

The program divides the full range of the spectrometer into 32 frequency bands and runs 32 experiments. This data is merged into one high-resolution data set. After enough data are collected, the Accept button becomes available. If ADC overflow occurs, a warning message appears.

8. After you see a strong signal (the signal will be stronger at low frequencies than at high frequencies) that does not cause ADC overflow, click the **Accept** button.
9. Attach a 50-ohm load:

- **UNITY^{INNOVA}** and **UNITY^{plus}** – Connect the 50-ohm load to the tune port.
 - **MERCURY-VX** – Connect the 50-ohm load to TUNE J5402 on the inside (probe side) of the magnet leg.
- Click the **50 Ohm Test** button.
The program divides the full range of the spectrometer into 32 frequency bands and runs 32 experiments. The data is merged into one high-resolution data set. After enough data are collected, the Accept button becomes available. If ADC overflow occurs, a warning message appears.
 - Click the **Accept** button and remove the 50-ohm load from the tune port.
 - After all three tests are finished, click the **Save** button.
The old calibration files are moved to .bak files, and new calibration files `std_ed.cal`, `std_es.cal`, and `std_er.cal` are calculated. These files are stored in `$vnmr/system/tune/tunecal`. If the files cannot be saved, the program produces a beep and an error message.
 - Click the **Close** button.

Restoring Previous Calibration Files

If you have an incorrect calibration file and do not want to re-run the calibration tests, restore the previous calibration files:

- Click the **Calibrate** button on the Tune Control Panel. The Tune Calibration window opens (see [Figure 15](#)).
- Click the **Restore Backup** button.

The program replaces the new calibration files with the .bak files. If the program does not find all of the backup files, an error message appears.

Restoring Original Calibration Files

System backup files can be installed by copying the following .cal files on top of the corresponding .sys files:

- `std_ed.cal` to `std_ed.sys`
- `std_es.cal` to `std_es.sys`
- `std_er.cal` to `std_er.sys`

To restore these system calibration files, click the **Restore Sys Backup** button. The new calibration files are replaced with the .sys calibration files created at system installation.

Obtaining Online Help

To get online descriptions of the buttons and fields in the Tune Control Panel, click the **Help** button with the right mouse button. A pull-down menu similar to that shown in [Figure 16](#) appears. Select the topic of interest from the menu.

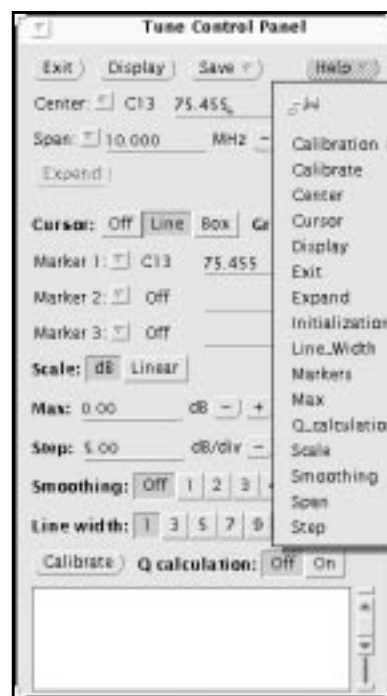


Figure 16. Help Pull-Down Menu

2.3 Tuning Probes with ^{UNITY}INOVA or UNITYplus

This section describes tuning probes with ^{UNITY}INOVA and UNITYplus spectrometers. Probes used with these systems can be tuned using one of two methods: using the TUNE INTERFACE or using the graphical qtune program. For instructions on tuning probes with MERCURY-VX, MERCURY, GEMINI 2000, UNITY, VXR, or Gemini spectrometers, refer to the specific tuning procedure later in this section.

Using the TUNE INTERFACE

^{UNITY}INOVA and UNITYplus systems use the TUNE INTERFACE for probe tuning:

- For 200-, 300-, and 400-MHz ^{UNITY}INOVA and UNITYplus spectrometers, the tune interface is located on the Magnet-Console Interface, as shown in [Figure 17](#).
- For 500-, 600-, and 750-MHz ^{UNITY}INOVA and UNITYplus spectrometers, the tune interface is located on the Dual-Preamplifier Assembly, as shown in [Figure 18](#).

The TUNE INTERFACE contains the following displays, readouts, and connectors:

- The liquid-crystal display at the top of the TUNE INTERFACE provides a digital and analog view of the tune level. The center part of the meter displays a three-digit number between 000 and 999 that indicates a probe tuning setting. The outer, analog display is an elliptical bar graph with a range of 0 to 1000. The analog display is useful for seeing a trend—if the signal is increasing or decreasing.
- Below the display are two single-digit readouts labeled CHAN and ATTEN. The CHAN switch on the tune interface activates the tuning function and sets the frequency to be tuned. Switch position 0 is for normal operation—no probe tuning. The other positions, 1 through 4, set the tuning frequency to that of the corresponding channel, and connect the output of channel 1's frequency synthesizer to the input of the tuning circuit. All probe tuning frequencies are generated by the synthesizer associated with channel 1. The ATTEN switch is active only when a channel has been selected and

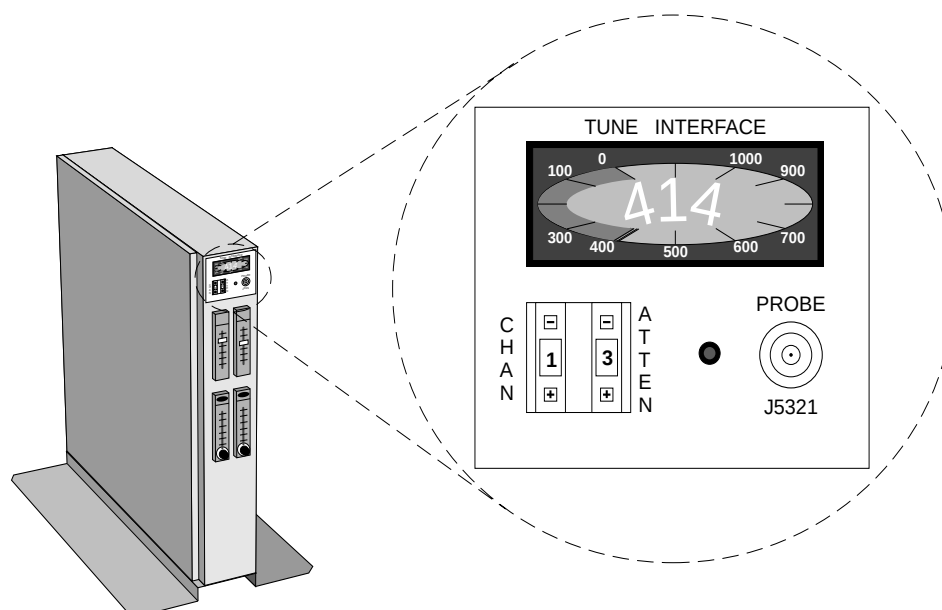


Figure 17. Tune Interface for ^{UNITY}INOVA and UNITYplus 200, 300, and 400 Systems

allows you to set the amount of attenuation. Attenuation is selected in units of 10 dB. The maximum attenuation is 79 dB, which is selected by setting of 8.

- At the lower right of the TUNE INTERFACE is a red indicator light and a BNC connector labeled PROBE J5321.

While in the tune mode, the system does not allow an acquisition to be started. Also, while the system is acquiring, the tune mode cannot be entered. When the tune mode is selected (CHAN is not set to 0), the Acquisition Status Window displays “Tuning” to indicate that tuning is in progress.

To Establish Tuning Frequencies and Channels

The ^{UNITY}INOVA and UNITYplus TUNE INTERFACE offers two methods for setting up tuning frequencies and channels—setting the experimental parameters (i.e., *tn*, *dn*, *dn2*, and *dn3*) or using the *tune* command.

Using Experimental Parameters

The experimental parameters are entered similar to the following example:

```
tn='H1' dn='13C' dn2='15n' su
```

In an experiment, the *tn*, *dn*, *dn2*, and *dn3* parameters correspond to channels 1, 2, 3, and 4, respectively. The required *su* command sets up the frequencies. If, however, any of these parameters are changed for tuning purposes, they must be restored before beginning acquisitions. If you do not want to disturb an experiment’s parameter set (and remember to change it back), use the *tune* command instead, described below.

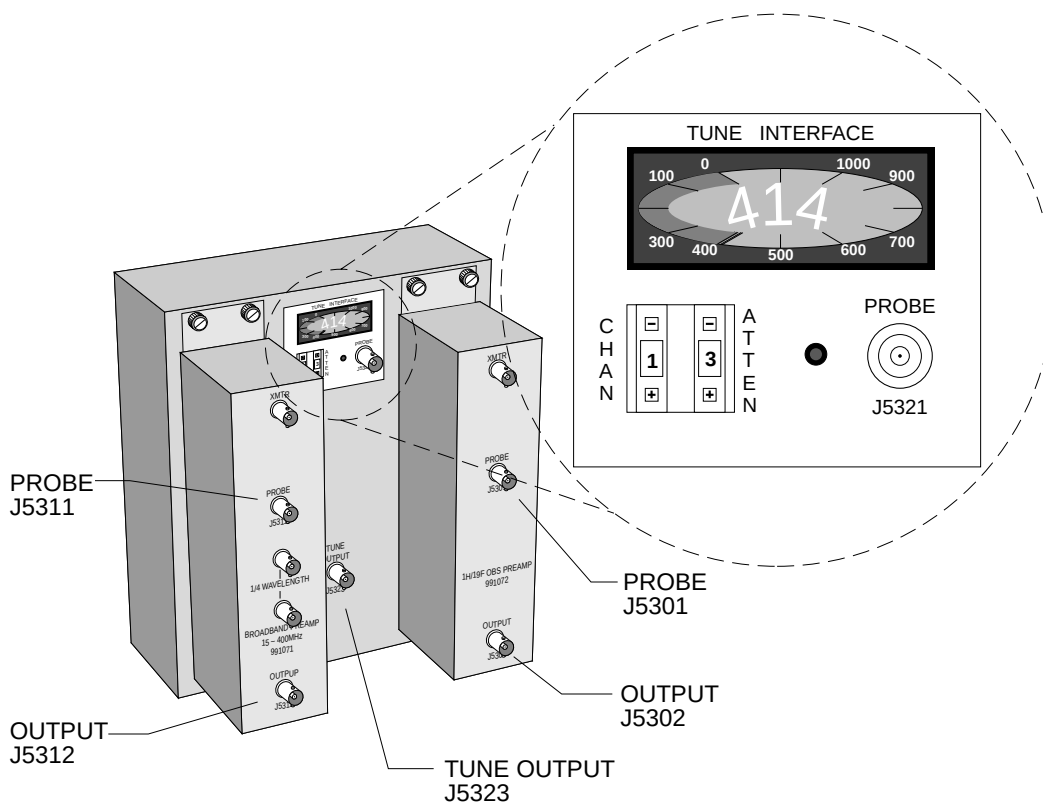


Figure 18. Tune Interface for ^{UNITY}INOVA and UNITYplus 500, 600, 750, and 800 Systems

Using the tune Command

The `tune` command is entered similar to the following example:

```
tune('H1', 'C13', 'N15')
```

The first argument ('H1') corresponds to channel 1, the second argument corresponds to channel 2, and so on. The `tune` command does not require the `SU` command.

If a desired frequency is not in the nucleus table, you can enter a frequency for a channel, similar to the following example:

```
tune('ch4', 'H1', 'ch3', 357.834)
```

In this example, ¹H is assigned to channel 4 and 357.834 MHz is assigned to channel 3. Alternatively, you can enter a string of the desired frequencies using the format:

```
tune(f1, f2, f3, f4)
```

where `f1` is the frequency, in MHz, for channel 1, `f2` is the frequency assigned to channel 2, and so on.

To Tune the Observe and Decouple Channels

After the tuning frequencies and channels have been established, use the following procedure to tune the probe.

1. Move the rf cable attached to the probe channel you want to tune using the appropriate step below. No filters should be in line during the tuning procedures:
 - ^{UNITY}INOVA and UNITYplus 200, 300, and 400 systems—disconnect the cable from the H Band CM or B Band CM port on the back of the magnet-console interface and connect it to the PROBE connector (J5321) on the TUNE INTERFACE, which is on the front of the magnet-console interface shown in [Figure 17](#).
 - ^{UNITY}INOVA and UNITYplus 500, 600, and 750 systems—disconnect the cable from the PROBE port on the broadband preamplifier (J5311) or the ¹H/¹⁹F preamplifier (J5301) and connect it to the PROBE port (J5321) on the TUNE INTERFACE, which is located on the Dual Preamp assembly shown in [Figure 18](#). Then disconnect the cable from the OUTPUT connector (J5312 or J5302) and connect it to the TUNE OUTPUT connector (J5323).

The LED flashes to indicate that the tune configuration is not yet correct.

2. Set the CHAN switch to the number of the channel to be tuned—1, 2, 3, and so on. The LED turns off. The tuning meter is now activated and should display a number in the digital display and a deflection on the analog display. The ATTEN switch setting can be changed at this point to increase or decrease the meter reading. The setting displayed in the center part of the meter can be reset later, permitting a probe to be retuned while repeating its PW90 value.
3. Tune the probe with the tuning controls on the probe. Refer to the section in this manual pertaining to your specific probe for tuning ranges and the capacitor or inductor sticks required for certain nuclei.

As the probe approaches being tuned, the number in the center of the liquid crystal display decreases. Tune the probe until the number is as close to 0 as possible. To facilitate tuning in the future, write down the channel and attenuator switch settings and tuning voltage.
4. When the probe is tuned, return the setting on the CHAN switch to 0 (the LED flashes and the liquid crystal display turns off).

5. Reconnect the rf cables in their original positions using the appropriate step below:
 - UNITY *INOVA* and UNITY *plus* 200, 300, and 400 systems—return the cable connected to the TUNE PROBE connector (J5321) to the appropriate connector on the back of the magnet-console interface (H Band CM or B Band CM).
 - UNITY *INOVA* and UNITY *plus* 500, 600, and 750 systems—return the cable connected to the TUNE PROBE connector (J5321) to the appropriate PROBE connector (J5311 or J5301). Then return the cable from the TUNE OUTPUT connector (J5323) to the appropriate OUTPUT connector (J5312 or J5302).

The LED is now off.

To Tune the Lock Circuit

The lock circuit of the probe is factory-tuned and does not require tuning in normal use. Follow the procedure below only if you have reason to believe the lock sensitivity has degraded or after you have intentionally detuned the lock circuit. To gain access to the lock tuning capacitors, the probe must be removed from the magnet *after the sample is ejected*. Figure 19 shows two different layouts of the lock tune and match capacitors.

1. Remove the sample from the probe.
2. Remove the probe from the magnet.
3. Disconnect the probe cable from the Lock Preamplifier connector (P5205 in the Magnet/Console Interface) and connect it to the PROBE connector (J5321).
4. On 500-, 600-, and 750-MHz systems, disconnect the cable from the OUTPUT connector (J5312 on the broadband preamp) and connect it to the TUNE OUTPUT connector (J5323).

The LED flashes to indicate the tuning setup is not yet complete.

5. Enter the following command exactly as shown (do not use single quotes around the argument):

tune(lockfreq)

This sets the tuning circuit to the lock frequency.

6. Set the CHAN switch to 1. The LED turns off.

The TUNE INTERFACE meter turns on and displays a number in the digital display and a deflection on the analog display. You can change the ATTEN switch setting to increase or decrease the meter reading.

7. Tune the lock circuit to the appropriate frequency by repeatedly tuning the two variable capacitors accessed through two small holes in the outer shield of the probe.

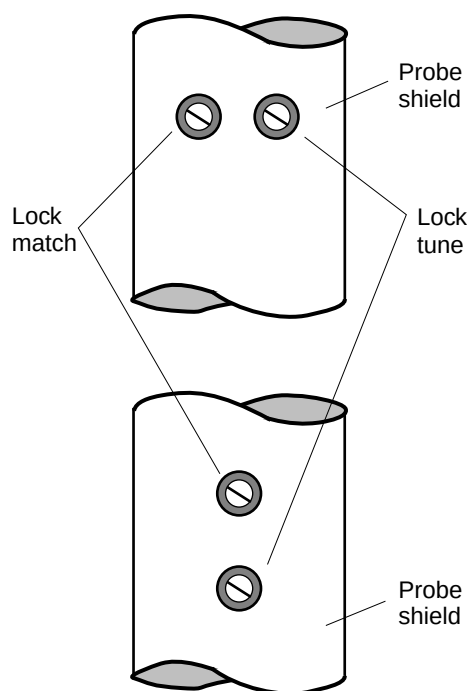


Figure 19. Lock Tune and Match Capacitors Locations

8. After the lock circuit is tuned, return the CHAN switch to 0 (the LED begins flashing and the TUNE INTERFACE display turns off).
9. Disconnect the cable from the PROBE connector (J5321) and return it to the Lock Preamplifier (P5205 in the Magnet/Console Interface). The LED turns off.
10. On 500-, 600-, and 750-MHz systems, disconnect the cable from the TUNE OUTPUT connector (J5323) and return it to the OUTPUT connector (J5312).
11. Reinsert the probe into the magnet.

2.4 Tuning Probes with *MERCURY-Series* or *GEMINI 2000* Systems

These procedures describe how to tune probes used with *MERCURY-series* or *GEMINI 2000* spectrometer systems. The connectors used to tune to probe are located on the back and inside of the left magnet leg and are illustrated in **Figure 23**.

MERCURY-VX systems can use the graphical qtune program for probe tuning, which is described in Section 2.2, "Using the qtune Graphical Probe Tuning Program," on page 25.

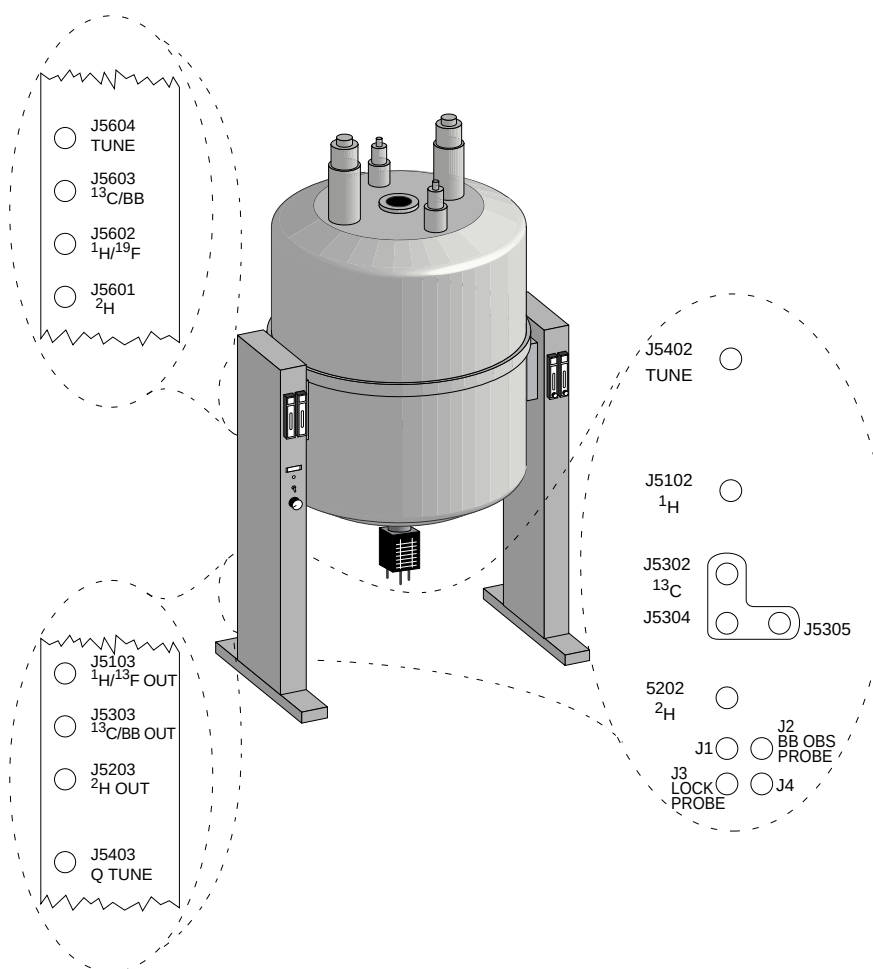


Figure 20. Connectors Used for Tuning *MERCURY* or *GEMINI 2000* Probes

Tuning the High-Band Observe and Decouple Channel

1. Join an appropriate experiment and enter **tn='xx' su**, where xx is the nucleus you want to observe, for example, **tn='H1'** for ^1H or **tn='F19'** for ^{19}F .
If the nucleus is not in the nucleus table, use **sfrq** instead of **tn**. Set **sfrq** to the nearest frequency you want to observe, and then type **su**. Once the correct frequency is determined, you can tune the probe.
2. On the inside of the left magnet leg, locate the cable connected to the ^1H (J5103) connector. Disconnect this cable and connect to the TUNE (J5402) connector.
3. On the back of the magnet leg, locate the cable connected to the $^1\text{H}/^{19}\text{F}$ (J5602) connector. Disconnect this cable and connect it to the TUNE (J5604) connector.
4. Turn on the transmitter with the appropriate command:
 - $^1\text{H}/^{13}\text{C}$ GEMINI 2000, enter **htune**.
 - 4-nucleus or broadband MERCURY-series or GEMINI 2000 enter **btune**.
5. Set the meter switch to the TUNE position. Adjust the TUNE knob for a mid-range reading.
6. Alternately adjust the tuning controls and the match control to obtain a minimum reading on the PROBE TUNING meter. Adjust the two tune controls so that each tune capacitor has an equal capacitance. Adjust the TUNE LEVEL attenuator or 10-position knob on the magnet leg to keep the PROBE TUNING meter in the lower half of the meter scale.
7. After a minimum has been obtained, enter **tunecoff** to turn off the transmitter.
8. Return the cable from the TUNE (J5604) connector to the $^1\text{H}/^{19}\text{F}$ (J5602) connector, return the cable from the TUNE (J5402) connector to the ^1H (J5102) connector, and set the tune meter switch back to SPIN.

Tuning the Low-Band Observe Channel

1. Join an appropriate experiment and enter the appropriate value for **tn**:
 - $^1\text{H}/^{13}\text{C}$ systems, enter **tn='C13' su**.
 - 4-nucleus or broadband systems, enter **tn='xx' su**, where xx is the nucleus you want to observe, for example, **'C13'** for ^{13}C or **'P31'** for ^{31}P .
If the nucleus is not in the nucleus table, use **sfrq** instead of **tn**. Set **sfrq** to the nearest frequency you want to observe, and then enter **su**. Once the correct frequency is determined, you can tune the probe.
2. Move the cable from the ^{13}C connector (J5302) to the TUNE connector (J5402).
3. For broadband systems, make sure the correct 1/4-wavelength cable is attached.
4. Move the cable from the ^{13}C connector (J5603) on the rear of the magnet leg to the TUNE connector (J5604) just above it.
5. Turn on the transmitter board for tuning with the appropriate command:
 - $^1\text{H}/^{13}\text{C}$ GEMINI 2000, enter **ctune**.
 - 4-nucleus or broadband MERCURY-series or GEMINI 2000, enter **btune**.
6. Place the meter switch in the TUNE position. Adjust the TUNE control knob for a mid-range reading.
7. Tune the probe using the appropriate step:

- For $^1\text{H}/^{19}\text{F}$ probes—turn the ^{13}C coil tuning (green) and ^{13}C coil matching (blue) controls to obtain a minimum tuning meter reading as follows: Turn the green control until meter reads a minimum reading. Then turn the blue control for a minimum meter reading. Adjust the TUNE knob if needed. Switch back and forth between the green and blue controls until you achieve an absolute minimum meter reading. If the tuning is far off, it may be better to turn each control past the minimum meter reading before turning the other control.
 - For broadband and switchable probes—alternately adjust the tuning controls and the match control to obtain a minimum reading on the PROBE TUNING meter. Adjust the two tune controls so that each tune capacitor has an equal capacitance. Adjust the TUNE LEVEL attenuator or 10-position knob on the magnet leg to keep the PROBE TUNING meter in the lower half of the meter scale.
8. Once a minimum has been obtained, enter **tuneoff** to turn off the transmitter.
 9. Return the two cables to their original positions and switch the tune meter switch back to SPIN.

Tuning the ^2H Lock Channel

The lock circuit is factory-tuned and in normal use will not require retuning. Follow the procedure below only if you have reason to believe the lock sensitivity has degraded or after you have intentionally detuned the lock circuit. To gain access to the lock tuning capacitors, the probe must be removed from the magnet after the sample is ejected.

1. Remove the sample, then remove the probe from the magnet.
2. Disconnect the lock cable from the magnet leg. Connect a cable from the LOCK PROBE connector to the TUNE connector (J5402) on the left magnet leg.
3. Connect the ^2H cable to the TUNE connector (J5604) on the back of the left magnet leg.
4. Enter **dtune**. This turns on the lock transmitter for tuning.
5. Move the TUNING meter switch to the TUNE position.
6. Tune the lock circuit to the proper frequency by alternately adjusting the two variable capacitors accessed through the two holes in the probe shield as shown in [Figure 21](#). Continue until a minimum reading is obtained on the TUNING meter.
7. Enter **tuneoff** to turn off the lock transmitter.
8. Return both cables to the original positions and switch the meter back to the SPIN position.

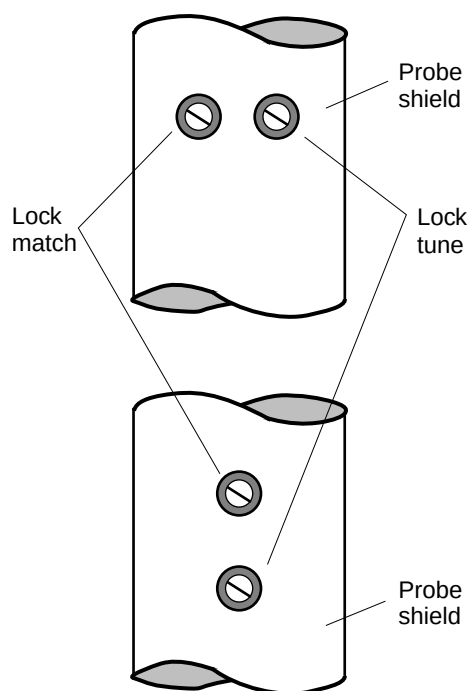


Figure 21. Lock Tune and Match Capacitors Locations

2.5 Tuning Probes with UNITY or VXR

Use the following procedures to tune probes used with UNITY and VXR spectrometer systems.

Tuning High-Band Observe and Decouple Channels

1. Enter the value for the transmitter nucleus parameter by entering **tn='xx' su**, where **xx** is the description of the desired observe nucleus, for example, **tn='F19' su** or **tn='H1' su**.
2. Move the rf cable from the high band channel you want to tune using the appropriate step below. Do all tuning without filters in line:
 - 200-, 300-, and 400-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the 1H/OBS/DEC port on the magnet leg and connect it to the tune port on the magnet leg.
 - 200-, 300-, and 400-MHz systems with a broadband preamplifier—Disconnect the cable from the H Band port on the switching relay or the 1H/OBS/DEC port on the magnet leg and connect it to the TUNE port on the magnet leg.
 - 500-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the PROBE port on the preamplifier and connect it to the TUNE port on the magnet leg.
 - 500- and 600-MHz systems with a dual preamplifier—Disconnect the cable from the PROBE port (J5301) and connect it to the TUNE PROBE port (J5321). Disconnect the cable from the OUTPUT port (J5302) and connect it to the TUNE OUTPUT (J5323).
 - 500- and 600-MHz decoupler—Disconnect the cable from the band pass filter on the side of the magnet leg and connect it to the TUNE PROBE port (J5321). Disconnect the cable from the OUTPUT port (J5302) and connect it to the TUNE OUTPUT port (J5323).
3. Turn the tuning selector to the OBS position and adjust the TUNE LEVEL switch to produce a signal on the PROBE TUNING meter.
 This routes a continuous wave (CW) observe transmitter signal to the observe coil. Unless the probe is perfectly tuned, the meter should register some deflection after a momentary delay. At this point, the transmitter light on the console or the remote status unit (as appropriate for your console) turns on. The receiver gain overload light may come on also.
4. Refer to the section in this manual that covers your probe for details on what tuning controls to adjust for optimum tuning and for tuning ranges.
5. Return the rf cables to the original positions using the appropriate step below:
 - 200-, 300-, and 400-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the TUNE port on the magnet leg and connect it to the 1H/OBS/DEC port on the magnet leg.
 - 200-, 300-, and 400-MHz systems with a broadband preamplifier—Disconnect the cable from the TUNE port on the magnet leg and connect it to the H Band port on the switching relay or the 1H/OBS/DEC port on the magnet leg.
 - 500-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the TUNE port on the magnet leg and connect it to the PROBE port on the preamplifier.

- 500- and 600-MHz systems with a dual preamplifier—Disconnect the cable from the TUNE PROBE port (J5321) and connect it to the PROBE port (J5301). Disconnect the cable from the TUNE OUTPUT (J5323) and connect it to the OUTPUT port (J5302).
- 500- and 600-MHz decoupler—Disconnect the cable from the TUNE PROBE port (J5321) and connect it to the band pass filter on the side of the magnet leg. Disconnect the cable from the TUNE OUTPUT port (J5323) and connect it to the OUTPUT port (J5302).

Tuning Low-Band Observe and Decouple Channels

1. Enter the value for the transmitter nucleus parameter by entering **tn='xx' su**, where **xx** is the description of the desired observe nucleus, for example, **tn='C13' su** to set up ^{13}C .
2. If the nucleus you are using requires a capacitor or inductor stick, insert it into the access hole at the bottom of the probe. Slide it into the probe until it stops, rotate it two or three times until it seats. For the correct stick refer to the tuning range section of the probe you are using.
3. Remove all filters for the tuning operation.
4. Move the rf cable attached to the X Channel you want to tune using the appropriate step below. Remove all filters for the tuning operation.
 - 200-, 300-, and 400-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the NORMAL port on the magnet leg and connect it to the TUNE port on the magnet leg.
 - 200-, 300-, and 400-MHz systems with a broadband preamplifier—Disconnect the cable from the B Band port on the switching relay or the 1H/OBS/DEC port on the magnet leg and connect it to the TUNE port on the magnet leg.
 - 500-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the NORMAL port on the preamplifier (or from the DCPLR port on the switching relay, located on the side of the magnet leg) and connect it to the TUNE port on the magnet leg.
 - 500- and 600-MHz systems with a dual preamplifier—Disconnect the cable from the PROBE port (J5311) and connect it to the TUNE PROBE port (J5321). Disconnect the cable from the OUTPUT port (J5302) and connect it to the TUNE OUTPUT (J5323).
 - 500- and 600-MHz decoupler—Disconnect the cable from the band pass filter on the side of the magnet leg and connect it to the TUNE PROBE port (J5321). Disconnect the cable from the OUTPUT port (J5302) and connect it to the TUNE OUTPUT port (J5323).
5. Turn the tuning selector switch to the OBS position and adjust the TUNE LEVEL switch to produce a signal on the PROBE TUNING meter.
 This routes a continuous wave (CW) observe transmitter signal to the observe coil. Unless the probe is perfectly tuned, the meter should register some deflection after a momentary delay. At this point, the transmitter light on the console or the remote status unit (as appropriate for your console) turns on. The receiver gain overload light may come on also.
6. Refer to the section in this manual that covers your probe for details on what tuning controls to adjust for optimum tuning and for tuning ranges.

7. Return the rf cables to the original positions using the appropriate step below:
 - 200-, 300-, and 400-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the TUNE port on the magnet leg and connect it to the NORMAL port on the magnet leg.
 - 200-, 300-, and 400-MHz systems with a broadband preamplifier—Disconnect the cable from the TUNE port on the magnet leg and connect it to the B Band port on the switching relay or the 1H/OBS/DEC port on the magnet leg.
 - 500-MHz systems with a $^1\text{H}/^{19}\text{F}$ preamplifier—Disconnect the cable from the TUNE port on the preamplifier and connect it to the NORMAL port on the magnet leg (or to the DCPLR port on the switching relay, located on the side of the magnet leg).
 - 500- and 600-MHz systems with a dual preamplifier—Disconnect the cable from the TUNE PROBE port (J5321) and connect it to the PROBE port (J5311). Disconnect the cable from the TUNE OUTPUT (J5323) and connect it to the OUTPUT port (J5302).
 - 500- and 600-MHz decoupler—Disconnect the cable from the TUNE PROBE port (J5321) and connect it to the band pass filter on the side of the magnet leg. Disconnect the cable from the TUNE OUTPUT port (J5323) and connect it to the OUTPUT port (J5302).

Tuning the Lock Channel

The lock circuit of the probe is factory-tuned, and in normal use will not require tuning. Follow the procedure below only if you have reason to believe the lock sensitivity has degraded or after you have intentionally detuned the lock circuit. To gain access to the lock tuning capacitors, the probe must be removed from the magnet *after the sample is ejected* (see [Figure 22](#)).

1. Set the spectrometer to observe ^2H by entering **tn='H2' su**.
2. Remove the sample from the probe.
3. Remove the probe from the magnet.
4. Connect the rf cable using the appropriate step below:
 - For 200- through 500-MHz systems—Connect a cable from the LOCK port on the probe to the TUNE port.
 - For 500 and 600-MHz systems with a dual preamplifier—Connect a cable from the LOCK port on the probe to the TUNE PROBE port (J5321). Disconnect the cable from the OUTPUT port (J4302) and connect it to the TUNE OUTPUT port (J5323).
5. Turn the tuning selector switch to the OBS position and adjust the TUNE LEVEL switch to produce a signal on the PROBE TUNING meter.

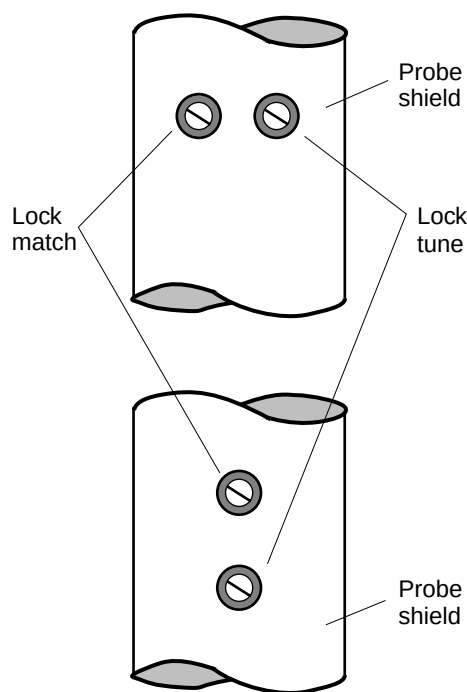


Figure 22. Lock Tune and Match Capacitors Locations

6. Tune the lock circuit to the optimum frequency by repeatedly tuning the two variable capacitors, accessed through two small holes in the outer shield of the probe, as shown in **Figure 22**.
7. Reconnect the lock channel.

2.6 Tuning Probes with Gemini Systems

These procedures describe how to tune probes used with Gemini spectrometer systems. The connectors used to tune to probe are located on the back and inside of the left magnet leg and are illustrated in **Figure 23**.

Tuning the High-Band Observe and Decouple Channel

1. Join an appropriate experiment and enter **TN=x SU**, where *x* is the atomic number for the nucleus you want to observe, for example, **TN=1 SU** for ^1H and **TN=19 SU** for ^{19}F . (If the nucleus is not in the nucleus table, type **NUDIS** to verify, and then use **SFRQ** instead of **TN**. Set **SFRQ** to the nearest MHz you want to observe, and then type **SU**. Once the correct frequency is determined, you can retune the probe.)

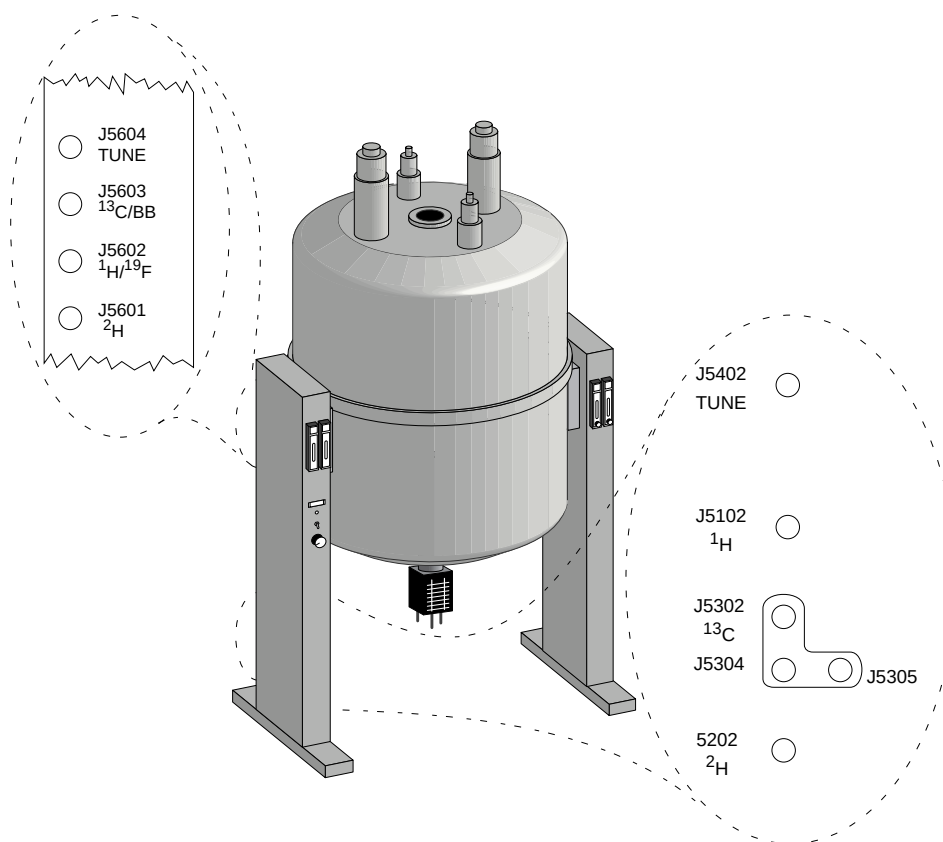


Figure 23. Connectors Used for Tuning Gemini Probes

2. On the inside of the left magnet leg, locate the cable connected to the ^1H (J5103) connector. Disconnect this cable and connect to the TUNE (J5402) connector.
3. On the back of the magnet leg, locate the cable connected to the $^1\text{H}/^{19}\text{F}$ (J5602) connector. Disconnect this cable and connect it to the TUNE (J5604) connector.
4. For a dual-frequency Gemini, enter HTUNE. For a broadband Gemini enter BTUNE. This turns on the transmitter for tuning.
5. Set the meter switch to the TUNE position. Adjust the TUNE knob for a mid-range reading.
6. Alternately adjust the tuning controls and the match control to obtain a minimum reading on the PROBE TUNING meter. Adjust the two tune controls so that each tune capacitor has an equal capacitance. Adjust the TUNE LEVEL attenuator (on the preamplifier housing) to keep the PROBE TUNING meter in the lower half of the meter scale.
7. Once a minimum has been obtained, type any character and press the Return key. This turns off the transmitter.
8. Return the cable from the TUNE (J5604) connector to the $^1\text{H}/^{19}\text{F}$ (J5602) connector, return the cable from the **TUNE** (J5402) connector to the ^1H (J5102) connector, and set the tune meter switch back to SPIN.

Tuning the Low-Band Observe Channel

1. Join an appropriate experiment and enter the value for TN as described in appropriate step below:
 - For $^1\text{H}/^{19}\text{F}$ systems, enter TN=13 SU.
 - For broadband systems, enter TN=x SU, where x is the atomic number for the nucleus you want to observe, for example, TN=1 SU for ^1H and TN=19 SU for ^{19}F .

If the nucleus is not in the nucleus table, type NUDIS to verify, and then use SFRQ instead of TN. Set SFRQ to the nearest MHz you want to observe, and then type SU. Once the correct frequency is determined, you can retune the probe.
2. Move the cable from the ^{13}C connector (J5302) to the TUNE connector (J5402).
3. Move the cable from the 13C connector (J5603) on the rear of the magnet leg to the TUNE connector (J5604) just above it.
4. For dual-frequency Gemini, enter CTUNE. For a broadband Gemini, enter BTUNE. This turns on the transmitter.
5. Place the meter switch in the TUNE position. Adjust the TUNE control knob for a mid-range reading.
6. Tune the probe using one of the steps below:
 - For dual-frequency probes—turn the ^{13}C coil tuning (green) and ^{13}C coil matching (blue) controls to obtain a minimum tuning meter reading as follows: Turn the green control until meter reads a minimum reading. Then turn the blue control for a minimum meter reading. Adjust the TUNE knob if needed. Switch back and forth between the green and blue controls until you achieve an absolute minimum meter reading. If the tuning is far off, it may be better to turn each control past the minimum meter reading before turning the other control.

- For broadband and switchable probes—alternately adjust the tuning controls and the match control to obtain a minimum reading on the PROBE TUNING meter. Adjust the two tune controls so that each tune capacitor has an equal capacitance. Adjust the TUNE LEVEL attenuator (on the preamplifier housing) to keep the PROBE TUNING meter in the lower half of the meter scale.
7. Once a minimum has been obtained, type any character and press the RETURN key. This turns off the transmitter.
 8. Return the two cables to their original positions and switch the tune meter switch back to SPIN.
 9. For broadband systems, make sure the correct 1/4-wavelength cable is attached.

Tuning the ^2H Lock Channel

The lock circuit is factory-tuned and in normal use will not require retuning. Follow the procedure below only if you have reason to believe the lock sensitivity has degraded or after you have intentionally detuned the lock circuit. To gain access to the lock tuning capacitors, the probe must be removed from the magnet *after the sample is ejected*.

1. Remove the sample, then remove the probe from the magnet.
2. Disconnect the lock cable from the magnet leg. Connect a cable from the LOCK PROBE connector to the TUNE connector (J5402) on the left magnet leg.
3. Connect the ^2H cable to the TUNE connector (J5604) on the back of the left magnet leg.
4. Enter DTUNE. This turns on the lock transmitter for tuning.
5. Move the TUNING meter switch to the TUNE position.
6. Tune the lock circuit to the proper frequency by alternately adjusting the two variable capacitors accessed through the two holes in the probe shield as shown in **Figure 24**. Continue until a minimum reading is obtained on the TUNING meter.
7. Type any character and press the RETURN key. This turns off the lock transmitter.
8. Return both cables to the original positions and switch the meter back to the SPIN position.

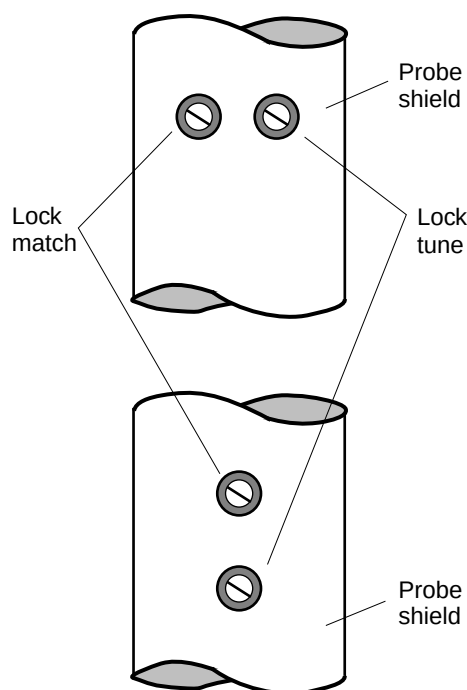


Figure 24. Lock Tune and Match Capacitors Locations

2.7 Setting the Lock Frequency on 600-MHz Systems

The potential for long-term drift of the Oxford 600-MHz magnet necessitates extending the frequency range of the lock channel.

1. Set the thumbwheels on the Offset Synthesizer board to 1500, and control the lock frequency using PTS frequency synthesizer #3 in the third cabinet.
2. Set the front panel knobs on the PTS to whatever setting is required to bring the lock signal on resonance.
3. The nominal setting for the synthesizer is 153.845000 MHz. Type this (or whatever the final setting turn out to be) using the `lockfreq` parameter; for example:
`lockfreq=153.845000`
All internal frequency calculations will be appropriately adjusted.

2.8 Changing Samples or Solvents

Check the reading on the probe tuning meter when changing samples, when changing the solvent at the same observe frequency, or when changing to a different observe frequency. The tuning is sensitive to changes in the dielectric constant and ionic strength of the sample.

In severely lossy samples (for example, 0.1 M aqueous NaCl), the 90° pulse width can increase dramatically. Check the pulse width with a similar sample doped with an observable signal and determine a new value for the pulse width. This permits a more rational choice of flip angle based on T_1 , the repetition rate, and the need for quantization.

Because the probe is a high-Q device, a lossy sample not only changes the value of the 90° pulse width but also degrades the sensitivity even when properly tuned. The change in sensitivity roughly parallels the change in 90° pulse width; that is, a doubling of the 90° pulse width results in about a 50-percent loss in sensitivity. Keep this factor in mind when choosing solvents.

With regard to sensitivity alone, a nonconducting or non-polar solvent is preferred. Electrical conductivity of the sample is the primary contributor to its lossy character and thus its probe detuning character.

2.9 Testing a Probe

The basic steps for testing a probe:

1. Create a probe calibration file as described in Section 2.10, “Using Probe Calibration Files,” this page.
2. Follow the appropriate test procedures in the *Acceptance Test Procedures* manual that shipped with your system or probe.
3. After the appropriate tests are completed for the probe, print the corresponding probe calibration file by entering the following command in the VNMR input window:

ACreport

2.10 Using Probe Calibration Files

VNMR includes probe calibration files in the directory `/vnmr/probes`. Probe calibration files contain `pw90`, `tpwr`, `dmf`, `dpwr`, and `pwX` settings for each probe used with the system. *GLIDE*, automation, and manual operation using macros—including `h1`, `hcosy`, and `dept`—retrieve appropriate power levels, the `pw90` value, and the `dmf` value

from the probe calibration file, instead of using preset values in the parameters files.

Successful operation depends on the existence of a probe calibration file for each probe that is used with the system.

When the *MERCURY*-series NMR spectrometer is installed, the proper probe calibration files are set up, so that the system is ready to use when the installer leaves the site.

The probe calibration files must be created when the system is installed or the first time a probe is used. Create or change the probe calibration file as follows:

To Create a Probe Calibration File

Do these steps for each probe that is tested.

1. Log in as `vnmr1`.
2. Enter the following command in the VNMR input window:

`addprobe('probe_name', 'system')`

Where *probe_name* is a descriptive name for the probe. For example, `sw5mm` for a 5-mm switchable probe, or `4nucPFG` for a 4-nucleus PFG probe. The `'system'` argument makes the probe calibration files for this probe available to all users by placing the files in `/vnmr/probes`. Without the argument, the files are placed in the `vnmr sys` directory of the user who entered the command (e.g., `~vnmr1/vnmr sys/probes`) and are only available to that user.

The values for power, `pw90`, `dmf`, and `pp` are initially set to 0 and filled in later by running the calibration procedures for ^1H , ^{19}F , ^{31}P , and ^{13}C using *GLIDE*.

To Change to a Different Probe Calibration File

After the probes are tested and calibrated, use this procedure to switch to the appropriate calibration file when a different probe is installed.

1. Log in as `vnmr1`.
2. Enter the following command in the VNMR input window;

`probe='probe_name'`

Where *probe_name* is the name of the directory in `/vnmr/probes` named for the new probe (e.g., `sw5mm`).

Chapter 3. **Probes for Liquids NMR**

Sections in this chapter:

- 3.1 “¹H/¹³C Computer-Switchable RT Probes” page 50
- 3.2 “AutoSwitchable Probes” page 52
- 3.3 “Broadband Probes” page 67
- 3.4 “Indirect Detection Probes” page 74
- 3.5 “Switchable Probes” page 82
- 3.6 “Triple-Resonance Probes” page 87

Each Varian liquids NMR probe is covered in a separate section of this chapter. Covered in the section are connecting the cables, tuning the probe, tuning ranges, and any special information specific to the probe.

3.1 $^1\text{H}/^{13}\text{C}$ Computer-Switchable RT Probes

This section covers the following $^1\text{H}/^{13}\text{C}$ computer-switchable, room-temperature probes for *MERCURY*-series, *GEMINI 2000* or Gemini systems:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Probe Part No.</i>
200	54	5	00-966128-00
300	54	5	00-966128-01

These probes can observe ^1H or ^{13}C without being removed from the magnet. Software controls switching by gating transmitters and preamplifiers at appropriate times. This section is organized as follows:

- “Connecting to *MERCURY-VX*, *MERCURY*, *GEMINI 2000*, or Gemini,” this page
- “Tuning Controls” page 51

CAUTION: Excessive heat can damage the sample, the decoupler coil and the tuning capacitors in the switchable probe. Reduce heat by limiting decoupler high power to 1.0 watts or less. Maintain an airflow of at least 9.5 liters per minute through the probe dewar to cool the sample.

Connecting to *MERCURY-VX*, *MERCURY*, *GEMINI 2000*, or Gemini

1. If the probe mounting plate has not been adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#).
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided.
3. Attach a ^1H notch filter to the ^{13}C connector (J5302) on the magnet leg. Connect a cable from the filter to the OBS (green) connector on the probe.
4. Make the following connections as appropriate for your system:
 - On 200-MHz systems, attach a second ^1H notch filter to the ^2H connector (J5202). Connect a cable from the notch filter to the LOCK (yellow) connector on the probe.
 - On 300-MHz systems, connect a cable from the ^2H connector (J5202) to the LOCK (yellow) connector on the probe.
5. Connect the decoupler cable proton observe from the ^1H connector (J5402) on the magnet leg to the DEC (red) connector on the probe.
6. Connect the cooling gas hose at the bottom of the probe housing to the cooling gas supply hose through the double-barbed connector supplied. This will provide cooling for the probe body and allows adjustment of temperature over a small range near room temperature if the supply gas temperature can be varied.

Tuning Controls

Computer-switchable probes have three controls that are operated by color-coded controls extending from the bottom of the probe:

<i>Function</i>	<i>Color</i>
^{13}C observe coil tuning	green
^{13}C observe coil matching	blue
^1H observe/decoupler coil tuning	red

The carbon observe coil tuning and matching controls are used to tune and match the transmitter/receiver coil in the probe to resonance at the observe transmitter frequency. The decoupler coil tuning control is used to tune the coil in the probe to resonance at the proton transmitter frequency. The probe may need to be tuned after a sample or solvent is changed. Refer to [Section 2.8, “Changing Samples or Solvents,” on page 46](#) for details.

3.2 AutoSwitchable Probes

AutoSwitchable probes combine simplified 4-nucleus operation with the versatility of multinuclear broadband nuclear observation. This section covers the following AutoSwitchable probes:

Magnet (MHz)	Bore (mm)	Sample (mm)	Range	Probe Part No.
300	54	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-903109-01
300 PFG	54	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-904083-01
300 LH	54	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-904086-01
300 PFG LH	54	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-904085-01
300	89	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-903110-01
300 PFG	89	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-904084-01
400 PFG	54	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-904089-01
400 PFG	89	5	$^1\text{H}/^{19}\text{F}/^{13}\text{C}/^{31}\text{P}$, $^1\text{H}/^{19}\text{F}/\{^{15}\text{N}-^{31}\text{P}\}$	01-904090-01

CAUTION: Do not attempt to tune the AutoSwitchable probe until you have thoroughly read this section. Any slight altering of the tuning can completely throw off the delicate balance between the two nuclei resonant on a port.

This section is organized as follows:

- “Connecting the Probe,” this page
- “Tuning Controls” page 54
- “Tuning the Probe on UNITYINOVA and UNITYplus” page 56
- “Tuning the Probe on MERCURY and GEMINI 2000” page 58
- “Tuning Ranges for AutoSwitchable Probes” page 62
- “Using Capacitor Sticks” page 63
- “Tuning the X-CHANNEL from an Unknown State to 4 NUC Mode” page 63
- “Calibrating the MERCURY Reflection Meter” page 64
- “Tuning the Low-Band Channel in Switchable Mode” page 64
- “Tuning Tables for 4-Nucleus to Switchable” page 66

Connecting the Probe

The following sections describe how to connect an AutoSwitchable probe to a system.

To Connect to ^{UNITY}INOVA, UNITYplus, UNITY, or VXR

1. If the probe mounting plate has not been adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#).
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the thumbscrews attached.
3. For PFG probes only, connect the PFG cable to the PFG connector on the probe base (see [Figure 25](#)).
4. Connect the observe cable according to one of the following:

- **300- and 400-MHz systems with the $^1\text{H}/^{19}\text{F}$ OBS preamplifier** – connect a low-pass filter and lock reject filter to the OBSERVE NORMAL connector on the magnet-console interface (magnet leg). Then connect a cable from the filter to the OBS connector on the probe.
 - **300- and 400-MHz systems with the broadband preamplifier** – connect a low-pass filter and lock reject filter to the B BAND connector or OBSERVE NORMAL connector on the magnet-console interface (magnet leg). Then connect a cable from the filter to the OBS connector on the probe.
5. Attach a second notch filter to the LK PROBE connector (J5205) on the lock preamplifier module inside the preamplifier housing. Connect a cable from the notch filter to the LOCK connector on the probe.
 6. Connect the high-band cable according to one of the following:
 - **300- and 400-MHz systems with the $^1\text{H}/^{19}\text{F}$ OBS preamplifier** – connect a high-pass filter to the 1H/OBS/DEC connector on the magnet-console interface (magnet leg). Then connect a cable from the filter to the $^1\text{H}/^{19}\text{F}$ connector (J6003) on the probe.
 - **300- and 400-MHz systems with the broadband preamplifier** – connect a high-pass filter to the H BAND connector or the 1H/OBS/DEC on the magnet-console interface (magnet leg). Then connect a cable from the filter to the $^1\text{H}/^{19}\text{F}$ connector (J6003) on the probe.

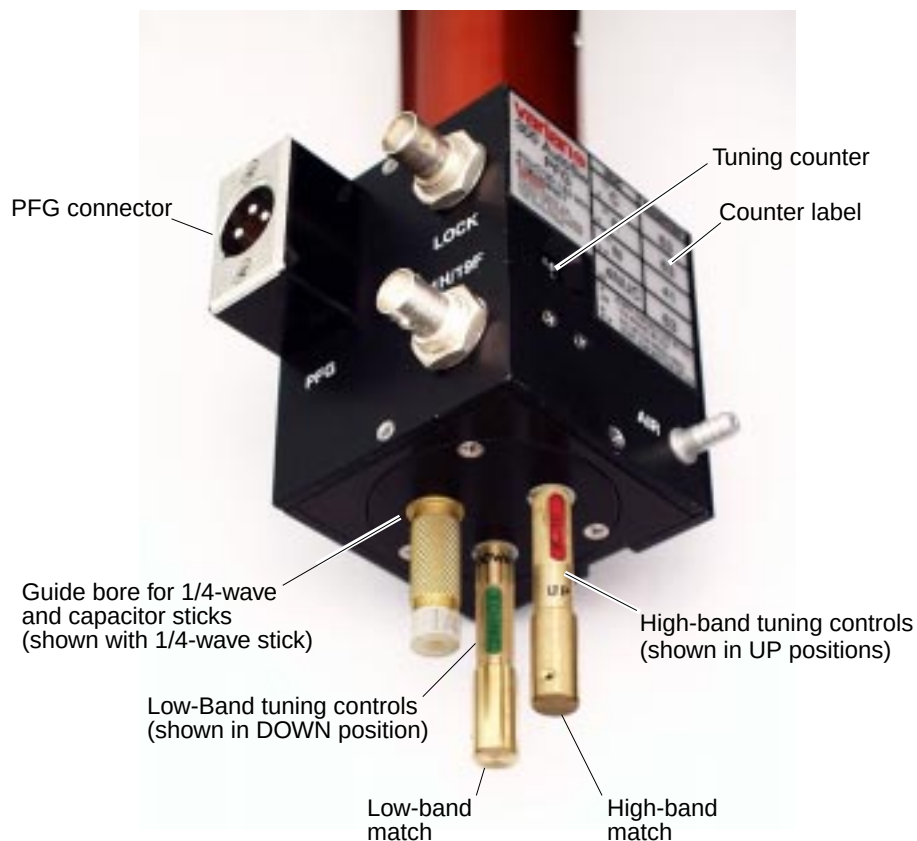


Figure 25. AutoSwitchable PFG Probe Tuning Controls And Connectors

7. Connect the cable from the variable temperature (VT) controller to the 9-pin connector (VT Control) on the probe.
8. Connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe.
9. Connect the body-cooling air hose to the hose barb protruding from the side of the probe and adjust the air flow to 10–15 LPM (refer to [Section 2.1, “Adjusting Spinning and Air Pressure,”](#) on page 24).

To Connect to MERCURY-Series, GEMINI 2000, or Gemini

1. If the probe mounting plate has not been adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,”](#) on page 18.
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the thumbscrews attached.
3. Attach the ^1H high-pass filter to the ^1H connector (J5102) on the magnet leg. Connect a cable from the filter to the $^1\text{H}/^{19}\text{F}$ connector on the probe.
4. Attach the low-pass filter to the ^{13}C connector (J5302) on the magnet leg (if the automation gradient relay is installed, use BB PRO J2). Connect a cable from the filter to the ^{13}C connector on the probe.
5. Connect a ^2H low-pass notch filter to the ^2H connector (J5202) on the magnet leg (if the automation gradient relay is installed, use LK PRO J3). Connect the cable to the LOCK connector on the probe.
6. For PFG probes only, connect the PFG cable to the PFG connector on the probe base (see [Figure 25](#)).
7. If the VT option is present, connect the cable from the VT controller to the 9-pin connector (VT Control) on the probe. Connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe.
8. Connect the body-cooling air hose to the hose barb (labeled AIR) on the probe and adjust the air to 10–15 LPM. (refer to [Section 2.1, “Adjusting Spinning and Air Pressure,”](#) on page 24).

Tuning Controls

The tuning controls for the AutoSwitchable probe extend from the bottom of the probe base, as shown in [Figure 25](#). The tuning controls of this probe are operated with the coaxial, brass-colored tuning knobs. Both the high-band channel (labeled 1H/19F in red) and the low-band channel (labeled X-CHANNEL in green) are tuned by independent controls that control two resonances for each channel.

The match function is controlled by the smooth knob on the bottom, while the knurled knob controls two tune functions: one in its DOWN position with the knob pushed all the way down until the word *DOWN* becomes visible and the gear system for the counter is engaged (for X-channel tuning), the other one in its UP position with the word *UP* fully visible and the gear system engaged.

High-Band Tuning Characteristics

[Figure 26](#) shows the tuning characteristics of the high-band tune controls. In the DOWN position, both resonances are moved simultaneously, with clockwise rotation of the knob

increasing the frequency. In the UP position, the frequency separation is changed, increasing the separation (while increasing the resonance frequency) by clockwise rotation.

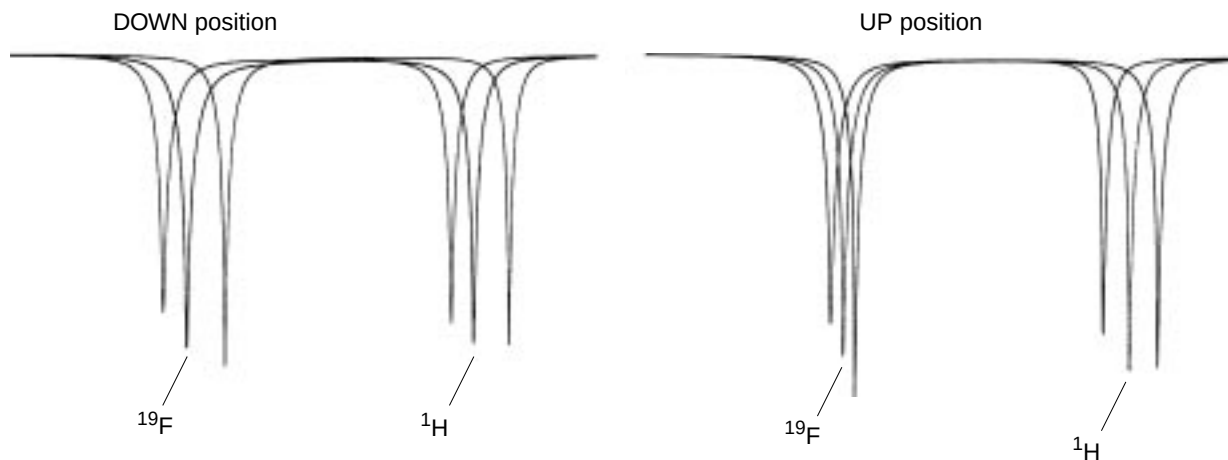


Figure 26. Tuning Characteristics of High-Band Tune Controls

Low-Band Tuning Characteristics in 4-Nucleus Mode

Figure 27 shows the tuning characteristics of the low-band tune controls. In the DOWN position, mainly the lower resonance frequency (^{13}C) is changed, with clockwise rotation of the knob increasing the frequency. In the UP position, mainly the upper resonance frequency (^{31}P) is changed, increasing the resonance frequency by clockwise rotation.

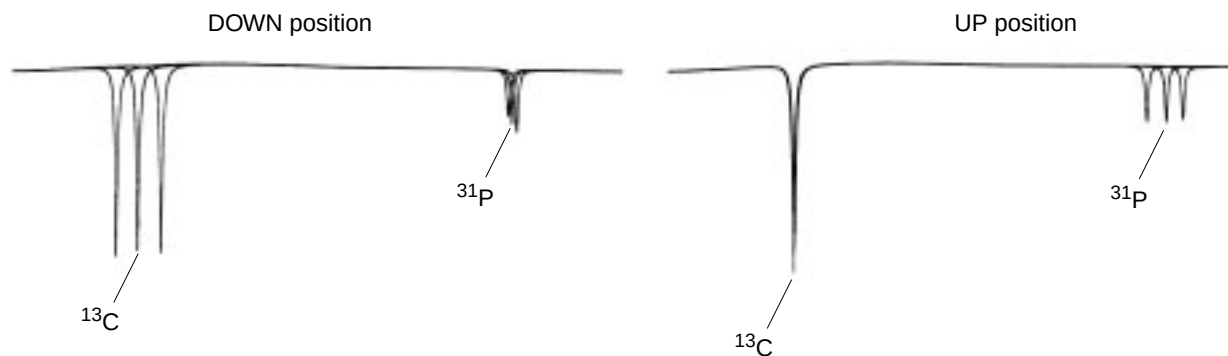


Figure 27. Tuning Characteristics of Low-Band Tune Controls

Tuning the Probe on ^{UNITY}INOVA and UNITY*plus*

This section contains the following procedures for tuning the AutoSwitchable probe on ^{UNITY}INOVA and UNITY*plus* systems:

- “To Tune the High-Band Channel (1H/19F),” this page
- “To Tune the Low-Band Channel in 4-Nucleus Mode (13C/31P)” page 57

To tune AutoSwitchable probes with *MERCURY* or *GEMINI 2000* systems, skip to “Tuning the Probe on *MERCURY* and *GEMINI 2000*” page 58.

An AutoSwitchable probe is shipped from the factory pretuned for a specific system. To obtain the best probe performance, tuning should be verified by running a pw90 calibration on the observe nucleus. Follow the procedures in this section to retune the probe.

Read the entire procedure before starting. Normally, the high-band channel does not require retuning and the broadband channel may need only a touchup.

To Tune the High-Band Channel (¹H/¹⁹F)

This section contains the procedures used to tune the AutoSwitchable probe high-band channel (¹H/¹⁹F) on ^{UNITY}INOVA and UNITY*plus* systems. Tuning the high-band channel requires preparing the NMR console and tuning the probe.

1. Make sure you have a sample with typical dielectric characteristics in the probe. Acetone is the recommended solvent for the tuning procedure to set up 4-nucleus mode for automated operation.
2. Connect the standard ¹H/¹⁹F/¹³C 1/4-wavelength cable.
3. Enter **s2pul**
4. Enter **dm='n' tn='H1' dn='F19' su**
This sets channel 1 (observe channel) to the ¹H frequency and channel 2 (decoupler channel) to the ¹⁹F frequency.
5. Connect the high-band connector of the probe (marked 1H/19F) to the TUNE connector on the magnet leg.
6. On the magnet leg, switch the channel selector to 2 (¹⁹F), and switch the attenuation selector to 6.
7. With the tune knob of the high-band channel (red label) in the DOWN position, tune the probe until the meter readout is minimum. Use the match knob to reduce the readout; repeat until the meter reads less than 50.
8. Switch the channel selector to 1 (¹H). Push the tune rod to the UP position, making sure the gear system inside the probe is engaged. Tune and match until the meter readout is minimized
9. Repeat steps 6 and 7.
10. Switch the attenuation selector to 7 and repeat steps 6 and 7.
11. Switch attenuation selection to 8 and repeat steps 6 and 7 until both channels are simultaneously less than 300.
12. Connect the probe back to the automation relay and switch the channel selector to 0.

Retuning the high-band channel is not required after sample changes or changes to the low-band channel. However, for highly lossy samples, the performance may improve if the high-band channel is tuned again.

To Tune the Low-Band Channel in 4-Nucleus Mode ($^{13}\text{C}/^{31}\text{P}$)

This procedure describes how to tune the AutoSwitchable probe low-band channel ($^{13}\text{C}/^{31}\text{P}$) in 4-nucleus mode on ^{UNITY}INOVA and ^{UNITY}plus. Tuning the low-band channel requires preparing the NMR console and tuning the probe.

In 4-nucleus mode, the AutoSwitchable probe is switchable between four nuclei by changing the frequency input in VNMR. No switching of the probe or the quarter-wavelength cable is required for switching frequencies. Each of the two ports on the probe is simultaneously tuned for two frequencies. The balance for each nucleus is set for optimum performance at the factory and requires no adjustment after basic tuning is achieved.

On systems equipped with only one PTS frequency synthesizer, the tuning nucleus must be changed in VNMR by changing the value of `tn`. For best results, follow the procedure “Tuning the X-CHANNEL from an Unknown State to 4 NUC Mode” on page 63.

1. Make sure you have a sample with typical dielectric characteristics in the probe. Acetone is the recommended solvent for the tuning procedure to set up 4-nucleus mode for automated operation.
2. Connect the standard $^1\text{H}/^{19}\text{F}/^{13}\text{C}$ 1/4-wavelength cable.
3. Enter **s2pul**.
4. Enter **dm='n' tn='P31' dn='C13' su**.
This sets channel 1 to ^{31}P and channel 2 to ^{13}C .
5. Connect the low-band connector of the probe (marked 13C/31P X CHANNEL) to the TUNE connector on the magnet leg.
6. Make sure the 1/4-wavelength stick is installed in the probe. Pull the tune rod of the X CHANNEL (green label) to the DOWN position, making sure that the gear system inside the probe is engaged. Set the counter to the value for 4-nucleus mode (refer to the label on your probe).
7. On the magnet leg, switch the channel selector to 2 (^{13}C), and switch the attenuation selector to 6.
8. Tune the probe until the meter readout is minimum. Use the Match knob to reduce the readout. Repeat until the meter reads less than 50.
9. Switch the channel selector to 1 (^{31}P). Push the tune rod in the UP position, making sure that the gear system inside the probe is engaged. Tune until meter readout is minimized. *Do not move the Match knob.*
10. Repeat the steps 7, 8, and 9.
11. Switch the attenuation selector to 7 and repeat steps 7, 8, and 9.
12. Switch the attenuation selector to 8. Repeat steps 7–9 until the reflection readouts are simultaneously less than 150 for channel 2 and less than 800 for channel 1.
Note that ^{31}P performance of this probe can be improved at the expense of ^{13}C performance if this ratio of reflection readouts is altered such that ^{31}P and ^{13}C have approximately the same reflected power.
13. Connect the probe back to the automation relay and switch the channel selector to 0.

Retuning the low-band channel in 4-nucleus mode is not required after sample changes. However, for highly lossy samples the performance may improve if the low-band channel is retuned for each sample.

Tuning the Probe on *MERCURY* and *GEMINI 2000*

This section contains the procedures for tuning the AutoSwitchable probe on *MERCURY* and *GEMINI 2000* systems.

- “To Tune the Highband Channel ($^1\text{H}/^{19}\text{F}$),” this page
- “To Tune the Low-Band Channel in 4-Nucleus Mode ($^{13}\text{C}/^{31}\text{P}$)” page 60

To tune the AutoSwitchable probe with *UNITYINOVA* or *UNITYplus* systems, Use the procedures in “Tuning the Probe on *UNITYINOVA* and *UNITYplus*” on page 56.

An AutoSwitchable probe is shipped from the factory pretuned for a specific system. To obtain the best probe performance, tuning should be verified by running a **pw90** calibration on the observe nucleus. Follow the procedures in this section to retune the probe.

Read the entire procedure before starting. Normally, the high-band channel does not require retuning and the broadband channel may need only a touchup.

To Tune the Highband Channel ($^1\text{H}/^{19}\text{F}$)

This section contains the procedures used to tune the AutoSwitchable probe high-band channel ($^1\text{H}/^{19}\text{F}$) on *MERCURY* and *GEMINI 2000* systems. Tuning the high-band channel requires preparing the NMR console and tuning the probe.

1. Move the probe and the transmitter cables as follows:
 - a. On the inside of the left magnet leg, locate the probe cable connected to the H1 (J5102) connector. Disconnect this cable and connect it to the TUNE (J5402) connector.
 - b. On the back of the magnet leg, locate the transmitter cable connected to the H1/F19 (J5602) connector. Disconnect this cable and connect it to the tune (J5604) connector.
2. Start by tuning H1:
 - a. Enter **tn='H1' su**.
 - b. Turn on the transmitter board for tuning:
On a $^1\text{H}/^{13}\text{C}$ *GEMINI 2000*, enter **htune**.
On a *MERCURY* or broadband *GEMINI 2000*, enter **btune**.
 - c. Set the meter switch to the TUNE position. Adjust the TUNE knob for a midrange reading on the TUNE meter.
 - d. Enter **tuneoff**.
3. Tune F19:
 - a. Enter **tn='F19' su**. Then enter **htune** or **btune**, depending on your system:
 $^1\text{H}/^{13}\text{C}$ *GEMINI 2000*, enter **htune**.
MERCURY or broadband *GEMINI 2000*, enter **btune**.
 - b. Check the TUNE METER reading.
 - c. Enter **tuneoff**.
4. Tune H1:
 - a. Set **tn='H1' su**. Then enter **htune** or **btune**, depending on your system:
 $^1\text{H}/^{13}\text{C}$ *GEMINI 2000*, enter **htune**.

- MERCURY* or broadband *GEMINI 2000*, enter **btune**.
- b. Check the TUNE METER reading for H1.
 - c. With the tune knob of the high-band channel (red label) in the DOWN position, tune the probe until the meter readout is minimum.
 - d. Enter **tuneoff**.
5. Tune F19:
- a. Enter **tn='F19' su**. Then enter **htune** or **btune**, depending on your system:
 $^1\text{H}/^{13}\text{C}$ *GEMINI 2000*, enter **htune**.
MERCURY or broadband *GEMINI 2000*, enter **btune**.
 - b. Check the TUNE METER. If the TUNE METER readings of both frequencies are significantly below the previous readings from steps 2 and 3, use both the UP and the DOWN positions to reduce the simultaneous reading to this lower value. *Do not use the match knob at this point.*
 - c. Enter **tuneoff**.
6. Verify specifications at this tuning level. Continue with the next steps only if the performance is insufficient.
7. Tune F19:
- a. Enter **tn='F19' su**. Then enter **htune** or **btune**, depending on your system:
 $^1\text{H}/^{13}\text{C}$ *GEMINI 2000*, enter **htune**.
MERCURY or broadband *GEMINI 2000*, enter **btune**.
 - b. With the tune knob of the high-band channel (red label) in the DOWN position, tune the probe until the meter readout is minimum. Turn the match knob to minimize the TUNE meter readout.
 - c. Alternate between the tune knob in the DOWN position and the match knob to obtain a minimum reading on the PROBE TUNING meter.
 - d. Adjust the TUNE LEVEL attenuation to keep the PROBE TUNING meter in the lower half of the meter scale.
 - e. After a minimum is obtain, enter **tuneoff** to turn off the transmitter.
8. Tune H1:
- a. Enter **tn='H1' su**.
 - b. Enter **htune** or **btune**, depending on your system:
 $^1\text{H}/^{13}\text{C}$ *GEMINI 2000*, enter **htune**
MERCURY or broadband *GEMINI 2000*, enter **btune**
 - c. With the tune knob of the high-band channel (red label) in the UP position, tune the probe until the meter readout is minimum. Turn the match knob to minimize the TUNE meter readout.
 - d. Alternate between the tune knob in the DOWN position and the match knob to obtain a minimum reading on the PROBE TUNING meter.
 - e. Enter **tuneoff**.

9. Repeat steps 3 through 8 until the PROBE TUNING meter reads mid-range or lower for H1 and F19, simultaneously, when the TUNE LEVEL attenuation is approximately at 3 o'clock.
Or, if the 10-position attenuator switch is installed, continue until the meter reading is less than 10 at switch position 6.
10. Return the transmitter and probe cable as follows:
 - a. Return the transmitter cable from the TUNE (J5604) connector to the H1/F19 (J5602) connector.
 - b. Return the probe cable from the TUNE (J5402) connector to the H1/F19 (J5102) connector.
11. Set the tune meter switch to SPIN.

To Tune the Low-Band Channel in 4-Nucleus Mode ($^{13}\text{C}/^{31}\text{P}$)

This procedure describes how to tune the AutoSwitchable probe low-band channel ($^{13}\text{C}/^{31}\text{P}$) in 4-nucleus mode on *MERCURY* and broadband *GEMINI 2000* systems. Tuning the low-band channel requires preparing the NMR console and tuning the probe.

For $^1\text{H}/^{13}\text{C}$ *GEMINI 2000* systems, using the probe in 4-nucleus mode is not possible. Instead, remove the 1/4-wavelength stick and use the 6.8 pF stick (red knob). Read this section for cabling instruction, and then follow the procedure for observing ^{13}C in the switchable mode, as described in “To Tune 300- and 400-MHz Probes to ^{31}P , ^{13}C , or ^{15}N ” on page 64.

In 4-nucleus mode, the AutoSwitchable probe is switchable between four nuclei by changing the frequency input in VNMR. No switching of the probe or the quarter-wavelength cable is required for switching frequencies. Each of the two ports on the probe is simultaneously tuned for two frequencies. The balance for each nucleus is set for optimum performance at the factory and requires no adjustment after tuning is achieved.

1. Connect the standard ^{31}P 1/4-wavelength cable.
2. Enter **s2pu1**.
3. Move the probe and transmitter cables as follows:
 - a. On the inside of the left magnet leg, locate the probe cable connected to the C13 (J5302) connector. Disconnect this cable and connect it to the TUNE (J5402) connector.
 - b. On the back of the magnet leg, locate the transmitter cable connected to the C13 (J5603) connector (if the automation gradient relay is installed, use BB PRO J2). Disconnect this cable and connect it to the tune (J5604) connector.
4. Check to see that the 1/4-wavelength stick is installed in the probe.
5. Set the tune meter switch to TUNE.
6. Tune C13:
 - a. Enter **tn='C13' su**.
 - b. Turn on the transmitter board for tuning:
For a $^1\text{H}/^{13}\text{C}$ *GEMINI 2000*, enter **ctune**.
For a *MERCURY* or a broadband *GEMINI 2000*, enter **btune**.
 - c. Set the meter switch to the TUNE position. Adjust the TUNE knob for a mid-range reading on the TUNE meter.

- d. With the tune knob of the low-band channel (green label) in the DOWN position, turn the knob until the counter is set to the value as shown on the label for 4 NUC mode. Turn the match knob to minimize the TUNE meter readout.
 - e. Adjust the TUNE LEVEL attenuation to keep the PROBE TUNING meter in the lower half of the meter scale.
 - f. After obtaining a minimum, enter **tuneoff** to turn off the transmitter.
7. Tune P31:
 - a. Enter **tn='P31' su**.
 - b. With the tune knob of the low-band channel (green label) in the UP position, tune the probe until the meter readout is minimum.
 - c. After obtaining a minimum, enter **tuneoff** to turn off the transmitter.
8. Repeat steps 6 and 7 until the PROBE TUNING meter reads mid-range on the TUNE METER for ^{13}C and ^{31}P when the TUNE LEVEL attenuation is approximately at 3 o'clock. Or, if the 10-position attenuator switch is installed, continue until the meter reading is less than 10 at switch position 6.
9. Tune C13 again:
 - a. Enter **tn='C13' su**
 - b. Turn on the transmitter board for tuning:
 For a $^1\text{H}/^{13}\text{C}$ GEMINI 2000, enter **ctune**.
 For a MERCURY or a broadband GEMINI 2000, enter **btune**.
 - c. Turn the match knob slowly to the right (counter-clockwise using the right-hand rule) until the TUNE METER reads between 30 to 40.
 - d. Enter **tuneoff**.
10. Return the transmitter and probe cables:
 - a. Return the transmitter cable from the TUNE (J5604) connector to the C13/P31 (J5603) connector.
 - b. Return the probe cable from the TUNE (J5402) connector to the C13/P31 (J5302) connector (if the automation gradient relay is installed, use BB PRO J2).
11. Set the tune meter switch to SPIN.

Tuning Ranges for AutoSwitchable Probes

The following tables list the tuning ranges for AutoSwitchable probes. Use these tables as a guide for choosing the appropriate capacitor or inductor sticks:

- For 300-MHz probes, see [Table 2](#).
- For 400-MHz probes, see [Table 3](#).

Table 2. Tuning Ranges for 300-MHz AutoSwitchable Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)*</i>
none	130–79 (³¹ P)
6.8 (red knob)	81–61 (¹³ C)
G 0 (blue knob)	63–49.8
G 18 (blue knob)	49.9–38.7
G 36 (blue knob)	38.8–32.8
G 51 (blue knob)	32.8–29.2 (¹⁵ N)
1/4-wave (gold knob)	4-nucleus (¹³ C/ ³¹ P)

* Probe ranges are approximate and valid for empty probe only. Ranges are slightly lower with a sample in a probe

Table 3. Tuning Ranges for 400-MHz AutoSwitchable Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)*</i>
none	170–93 (³¹ P, ¹³ C)
G 0 (blue knob)	95–45.8
G 18 (blue knob)	59–45.5
G 36 (blue knob)	46.2–39.5 (¹⁵ N)
1/4-wave (gold knob)	4-nucleus (¹³ C/ ³¹ P)

* Probe ranges are approximate and valid for empty probe only. Ranges are slightly lower with a sample in a probe

Using Capacitor Sticks

This procedure describes how to use the capacitor sticks, which extend the tuning range of the probe. Refer to [Table 2](#) and [Table 3](#) for tuning ranges.

1. Remove the capacitor stick from the protective shipping container.

The stick value is labeled at the bottom end of the stick. Notice that the new sticks are color coded for easier identification:

- red knobs – regular capacitor sticks (not required for all probes)
- blue knobs – grounded capacitor sticks
- gold knobs – 1/4-wavelength sticks

The top of the stick has a flat region that serves as a key for proper orientation of the capacitor stick in the probe.

2. Insert the stick into the probe bottom until you feel the tip of the stick hit a barrier.
3. Hold the plastic stick end (not the colored knob) and slightly rotate the stick until you feel the key engage and the stick move inward slightly.
4. Continue inserting the stick the rest of the way.
5. When the threads of the stick knob touch the bottom of the probe, twist the knob to affix the stick to the probe.

Tighten to finger-tight—overtightening could damage the probe.

Tuning the X-CHANNEL from an Unknown State to 4 NUC Mode

To tune the X channel from an unknown tuning state, you basically focus on ^{13}C while gently adjusting the match.

1. Make sure that the 1/4-wavelength stick is installed and the correct sample is inserted. Then set the counter (DOWN position) to the factory setting of 4 NUC (see counter label on the probe).
2. Set the console to ^{13}C , and turn only the match knob to minimize the reflection reading. The match capacitor should be only a few turns from minimum, turn it counter-clockwise until it hits the stop; then, turn it about 5 turns clockwise again.
3. Set the console to ^{31}P , turn only the UP position to get the reflection minimum; then, switch back to ^{13}C and turn only the match to minimize ^{13}C .
4. Set the console to ^{31}P and turn only the UP position to get the reflection minimum. This gets you close enough to both frequencies to get started.
5. Change back to ^{13}C , turn the DOWN position and match to optimize the reflection. The counter should remain fairly close to the factory-set value.
6. Switch to ^{31}P and turn only the UP position to minimize the reflection.
7. Repeat steps 5 and 6 once.
8. Add about a quarter-turn clockwise to match. Turn only the DOWN position to minimize ^{13}C ; turn only the UP position to minimize ^{31}P .

Do not adjust match anymore. The meter readout should be much better on ^{13}C than on ^{31}P —about 4 to 5 times better on UNITYINOVA or about 10 units on *MERCURY*.

Calibrating the *MERCURY* Reflection Meter

Calibrate the *MERCURY* reflection meter as follows.

1. Disconnect the cable from the tune connector (J5602).
2. Adjust the tune attenuation knob until the deflection is at the maximum meter readout.
3. Repeat this calibration after each switch in frequency
4. Write down the meter readout for both frequencies for future reference.
5. Reconnect the cable to the tune connector (J5602).

Tuning the Low-Band Channel in Switchable Mode

In switchable mode, the AutoSwitchable probe is similar in operation to any other switchable probe. The high-band port is simultaneously tuned to ^1H and ^{19}F , while the low-band port is tuneable from ^{15}N to ^{31}P . For best results, use the correct 1/4-wavelength cable and the standard filters (the ^1H high-pass for the $^1\text{H}/^{19}\text{F}$ channel and the lock-reject and low-pass filter for the $^{13}\text{C}/^{31}\text{P}/\text{X}$ channel).

Refer to the tuning ranges in [Table 2](#) and [Table 3](#) and select the appropriate capacitor stick. This section contains the following procedures:

- “To Tune 300- and 400-MHz Probes to ^{31}P , ^{13}C , or ^{15}N ,” this page
- “To Tune 300-MHz Probes to 63–130 MHz” page 65
- “To Tune 300-MHz Probes to 30–63 MHz” page 65
- “To Tune 400-MHz Probes to 94–170 MHz” page 65
- “To Tune 400-MHz Probes to 39–94 MHz” page 66

To Tune 300- and 400-MHz Probes to ^{31}P , ^{13}C , or ^{15}N

Use this procedure for tuning 300- and 400-MHz AutoSwitchable probes to ^{31}P , ^{13}C , or ^{15}N .

1. Set the spectrometer to observe the desired nucleus.
2. With the standard sample in the probe, tune as follows:
 - **^{31}P nucleus** – Use no capacitor stick. Preset the down tuner to the factory counter value. Use the UP position and match to find the reflection minimum. Match should be only a few turns from minimum (fully counterclockwise), preset match first, then use the UP position to find the resonance. Iteratively twist the UP position and match for optimum reflection.
 - **^{13}C nucleus, 300-MHz probes** – Use the 6.8 pF capacitor stick (red knob). Preset the counter to the factory value, leave match close to the 4-nucleus or ^{31}P setting, use UP position to find resonance. Use the UP position and match to optimize.
 - **^{13}C nucleus, 400-MHz probes** – Use no capacitor stick. Preset counter to factory value, leave match close to the 4-nucleus or ^{31}P setting, use UP position to find resonance. Use UP position and match to optimize.
 - **^{15}N nucleus** – Use the appropriate capacitor stick: G 51pF (blue knob) for 300-MHz probes or G 36pF (blue knob) for 400-MHz probes. Note that the UP position tuner has no effect with capacitor sticks with the blue knob (G-type), leave it in 4-nucleus position for easier return. Preset the counter to factory

value, add match (clockwise) to find resonance, use DOWN position and match to optimize.

3. Return cables to position prior to tuning.

To Tune 300-MHz Probes to 63–130 MHz

Use this procedure for tuning a 300-MHz AutoSwitchable probe to nuclei in the frequency range of 63–130 MHz.

1. Choose the appropriate capacitor stick (refer to [Table 2](#)):
 - 79–130 MHz, no stick
 - 63–79 MHz, 6.8 pF stick
2. Preset the Broadband match knob to only a few turns from minimum (fully counter-clockwise, then again several turns clockwise).
3. Preset the counter by turning the Broadband tune knob in the DOWN position to a value appropriate for the nucleus of interest. If you have operated your probe at this frequency before, refer to your notes. Otherwise find an approximate value by interpolating between 0 and the setting for ^{31}P for frequencies above 79 MHz or interpolating between 0 and 80 for frequencies in the range of 63–79 MHz.
4. Turn the Broadband tune knob in the UP position and the match knob to find the reflection minimum.
5. Repeat with the tune knob in the UP position and the match knob to minimize the reflection meter reading.
6. Record the counter reading for future reference.

To Tune 300-MHz Probes to 30–63 MHz

Use this procedure for tuning a 300-MHz AutoSwitchable probe to nuclei in the frequency range of 30–63 MHz.

1. Use the appropriate capacitor stick (refer to [Table 2](#)).
2. Turn the Broadband tune knob in the DOWN position to find the resonance. If you have operated your probe at this frequency before, refer to your notes for the appropriate counter setting.
 Note that the UP position of the tune knob has no effect with grounded capacitor sticks (blue knob), you can leave it in 4-nuc position for easier return to 4-nucleus operation.
3. Repeat with the tune knob in the DOWN position and the match knob to minimize the reflection meter reading.
4. Record the counter reading for future reference.

To Tune 400-MHz Probes to 94–170 MHz

Use this procedure for tuning a 400-MHz AutoSwitchable probe to nuclei in the frequency range of 94–170 MHz. Use no capacitor stick.

1. Preset the Broadband match knob to only a few turns from minimum (fully counter-clockwise, then again several turns clockwise).
2. Preset the counter by turning the Broadband tune knob in the DOWN position to a value appropriate for the nucleus of interest. If you have operated your probe at this

frequency before, refer to your notes. Otherwise find an approximate value by interpolating between the settings for ^{13}C and ^{31}P .

3. Turn the Broadband tune knob in the UP position and the match knob to find the reflection minimum.
4. Repeat with the tune knob in the UP position and the match knob to minimize the reflection meter reading.
5. Record the counter reading for future reference.

To Tune 400-MHz Probes to 39–94 MHz

Use this procedure for tuning a 400-MHz AutoSwitchable probe to nuclei in the frequency range of 39–94 MHz.

1. Insert the appropriate capacitor stick (refer to Table 3).
2. Turn the Broadband tune knob in the DOWN position to find the resonance. If you have operated your probe at this frequency before, refer to your notes for the appropriate counter setting.
Note that the UP position of the tune knob has no effect with grounded capacitor sticks (blue knob), you can leave it in 4-nuc position for easier return to 4-nucleus operation.
3. Repeat with the tune knob in the DOWN position and the match knob to minimize the reflection meter reading.
4. Record the counter reading for future reference.

Tuning Tables for 4-Nucleus to Switchable

This section provides a summary of tuning procedures for 4-nucleus to switchable operation. For 300-MHz probes, see Table 4. For 400-MHz probes, see Table 5

Table 4. 300-MHz AutoSwitchable Tuning Summary

<i>Nucleus</i>	<i>Match</i>	<i>Tune – UP</i>	<i>Tune–DOWN</i>	<i>Capacitor Stick</i>
^{31}P	clockwise	clockwise	counter ¹	none
^{13}C	clockwise	clockwise	counter	6.8 pF (red knob)
^{15}N	clockwise	(no effect) ²	counter	G51 pF (blue knob)

1. Counter settings are based on an empty probe.

2. Tuning in the up position does not affect the circuit when using grounded capacitor sticks.

Table 5. 400-MHz AutoSwitchable Tuning Summary

<i>Nucleus</i>	<i>Match</i>	<i>Tune – UP</i>	<i>Tune–DOWN</i>	<i>Capacitor Stick</i>
^{31}P	clockwise	clockwise	counter ¹	none
^{13}C	clockwise	counter-clockwise	counter	none
^{15}N	clockwise (~ 10 turns)	(no effect) ²	counter	G36 pF (blue knob)

1. Counter settings are based on an empty probe.

2. Tuning in the up position does not affect the circuit when using grounded capacitor sticks.

3.3 Broadband Probes

This section covers the following broadband probes:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Range</i>	<i>Probe Part No.</i>
200	54	5	20–81	00-969854-00
200	54	10	20–81	00-969847-00
200	89	10	20–81	00-969848-00
300	54	5	30–122	00-969841-00
300	54	10	30–122	00-969837-00
300	51	10	^{15}N – ^{31}P	01-902660-00
300	89	5	30–122	00-969842-00
300	89	10	^{15}N – ^{31}P	01-902657-00
400	51	10	^{15}N – ^{31}P	01-902244-00
400	89	10	^{15}N – ^{31}P	01-902260-00
500	51	5	^{15}N – ^{31}P	01-902289-00
500	51	10	^{15}N – ^{31}P	01-902646-00
600	51	5	^{15}N – ^{31}P	00-992554-00

This section is organized as follows:

- “Connecting to UNITY-Series or VXR,” this page
- “Connecting to MERCURY-Series, GEMINI 2000, or Gemini” page 68
- “Tuning the Broadband Probe” page 69
- “Returning to Normal Operation” page 70
- “Observing Protons” page 71
- “Using the Low-Frequency Broadband Probe” page 71
- “Tuning Ranges for Broadband Probes” page 72

Connecting to UNITY-Series or VXR

To connect a broadband probe to a ^{UNITY}INOVA, UNITY*plus*, UNITY, or VXR system:

1. If the probe mounting plate has not been adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#).
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided.
3. In the lock channel line, install the appropriate ^2H bandpass filter listed in the following table.

<i>Magnet (MHz)</i>	<i>^2H Bandpass Filter</i>
300	BE46-4-6BB
400	BE61-10-8BB
500	BE77-3.8-8BB
600	BE95-12-8BB

Filter model numbers describe the filter's function. For example, the 500-MHz bandpass filter model number is BE77-3.8-8BB, where the 77 indicates 77 MHz and "B" indicates bandpass—an "L" instead would indicate low-pass.

Expect the lock phase to change upon addition of the filter. Note that the ^2H bandpass filter can be left in the system at all times; however, it causes about 20% loss in lock sensitivity

4. Connect the observe channel according to the appropriate step below:
 - For the $^1\text{H}/^{19}\text{F}$ OBS preamplifier, attach a low-pass filter to the OBSERVE NORMAL or BB OBS/DCPL connector on the magnet-console interface (magnet leg). Connect a cable from the low-pass filter to the OBS connector on the probe.
 - For the broadband preamplifier, connect a proton frequency low-pass filter to the B BAND connector or the OBSERVE NORMAL on the magnet-console interface (magnet leg). Connect a cable from the low-pass filter to the OBS connector on the probe.
 - For the dual preamplifier, connect a low-pass filter to J5311 on the preamplifier. Connect a cable between the low-pass filter and the B BAND connector (K5008 NC). Connect a cable from the B BAND connector (K5008 CM) to the OBS connector on the probe.
5. Connect the decoupler channel according to the appropriate step below:
 - For the $^1\text{H}/^{19}\text{F}$ OBS preamplifier, connect a high-pass filter to the 1H/OBS/DEC or HB OBS/DCPL connector on the magnet-console interface (magnet leg). Connect a cable from the high-pass filter to the DEC connector on the probe.
 - For the broadband preamplifier, connect a high-pass filter to the H BAND or to the 1H/OBS/DEC connector on the preamplifier. Connect a cable from the high-pass filter to the DEC connector on the probe.
 - For the dual preamplifier, connect a cable between the bandpass filter located on the side of the preamplifier assembly and the DEC connector on the probe.

Connect a high-pass filter in series if the highband will be used as an observe channel.

6. Select an external quarter-wavelength cable to match the frequency of the observe nucleus. Attach this cable between the two BNC connectors labeled 1/4 WAVELENGTH on the preamplifier.
7. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.
8. Connect the nitrogen VT gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe. **Figure 3** shows a pneumatics connection diagram.
9. Connect the body-cooling air hose to the hose barb protruding from the bottom of the probe. **Figure 3** shows a pneumatics connection diagram.

Connecting to **MERCURY-Series, GEMINI 2000, or Gemini**

To connect a broadband probe to a **MERCURY-series, GEMINI 2000, or Gemini** system:

1. If the probe mounting plate has not been adjusted yet for the magnet, adjust the plate as described in **Section 1.3, "Installing the Probe and Upper Barrel," on page 18.**
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the thumbscrews provided.

3. Connect a cable and lowpass filter from the ^{13}C connector (J5302) on the magnet leg to the OBS connector on the probe.
4. Connect a cable and lowpass notch filter from the ^2H connector (J5202) to the LOCK connector on the probe.
5. Connect the appropriate quarter-wavelength cable on the broadband preamplifier. Connect the proper cable for the observe frequency, as indicated in the following table, to J5304 and J5305 on the left magnet leg.

Frequency (MHz)	Cable (Color)
44–178	Yellow
37–118	Black
11–45	Blue

6. Connect the decoupler/proton observe cable and highpass filter from the ^1H connector (J5102) to the DEC connector on the probe. Also connect a ^1H high-pass filter between the ^1H connector (J5102) and the coax cable.
7. If the VT option is present, connect the cable from VT controller to the 9-pin connector (VT Control) on the probe, and connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe.
8. Connect the body-cooling air hose to the hose barb on the bottom of the probe and adjust the air flow to optimize sample insertion, ejection, and spinning (refer to [Section 2.1, “Adjusting Spinning and Air Pressure,”](#) on page 24).

Tuning the Broadband Probe

For best probe performance, place the sample to be analyzed in the probe during tuning and follow the ^1H channel and X channel tuning procedures each time you change samples.

The broadband probe, pictured in [Figure 28](#), incorporates a numerical counter system and probe label on the broadband channel that corresponds to the tuning of the nuclei to be decoupled/observed. An example of the probe label is shown in [Figure 29](#). The knurled tune knob changes the values on the numerical counter, while the smooth knob operates the matching capacitor. The lower the frequency of the nucleus, the more the smooth match knob needs to be turned clockwise.

Follow the appropriate tuning procedures for your system:

- ^1H INOVA, UNITYplus – See [Section 2.3, “Tuning Probes with UNITY INOVA or UNITYplus,”](#) on page 33.
- MERCURY-series, GEMINI 2000 – See [Section 2.4, “Tuning Probes with MERCURY-Series or GEMINI 2000 Systems,”](#) on page 37.
- UNITY, VXR – See [Section 2.5, “Tuning Probes with UNITY or VXR,”](#) on page 40.
- Gemini – See [Section 2.6, “Tuning Probes with Gemini Systems,”](#) on page 43.

CAUTION: Do not force the tuning capacitors to turn past the end of their ranges. The end of the range is indicated by an increase in torque and not necessarily a stop to the rotation. Forced turning can cause serious mechanical damage to the probe, requiring factory repair.

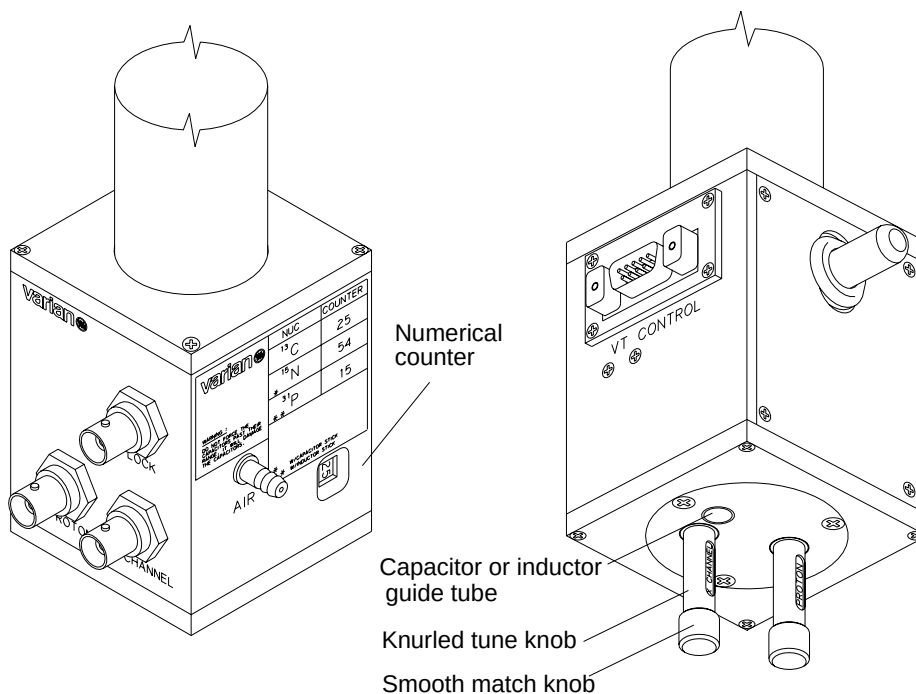


Figure 28. Broadband Probe

Tuning the ^1H Channel

Alternately adjust match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the red PROTON inset label turns the tuning capacitors, while the smooth lower knob turns the match capacitor (see Figure 28). When tuning to ^{19}F , turn the knurled knob counter-clockwise and adjust the match knob.

Tuning the X Channel

Turn the knurled X CHANNEL knob, shown in Figure 28, to a numerical counter value that corresponds to the nucleus to be observed. The probe label (see Figure 29) shows four popular nuclei. To fine tune, alternately adjust the smooth and knurled X CHANNEL match and tune knobs to find the minimum reflected power on the tune meter.

varian 600 BB 60 – 243 MHz 0099255400 WARNING: DO NOT FORCE THE CAPACITORS PAST THEIR RANGE. IT WILL DAMAGE THE CAPACITORS.	NUC	COUNTER
	^{13}C	08
	* ^{31}P	08
	** ^{15}N	40
	^{29}Si	32
* 8T INDUCTOR STICK ** 21PF CAPACITOR STICK		

Figure 29. Broadband Probe Label with Example Counter Values

Returning to Normal Operation

After tuning the probe, enter `dm='n' su` to make sure the decoupler is not inadvertently left on. Check ^1H channel tuning after X-channel tuning as well as after a sample change.

Observing Protons

Protons can be observed directly using the broadband probe by entering `tn='H1' su`. Usually no tuning is required because the decoupler coil is normally tuned to protons. The decoupler coil is longer and larger in diameter than the observe coil so that corresponding homogeneity and sensitivity changes are expected.

Excellent resolution is obtained with a 5-mm sample (with a 5-mm turbine) in the 10-mm probe. Sensitivity can be up to 50 percent of that achieved using the ^1H probe in an optimal manner. The 90° pulse width for protons is different and longer than that for the ^1H probe.

Recall a proton parameter set (or reset relevant parameters in an existing set) and begin acquisition. Going between broadband observe and proton observe and back takes seconds rather than the time required for probe changes. This can be very useful in VT experiments, where both proton and other nuclei spectra are required at each temperature, or on samples with a short lifetime.

Using the Low-Frequency Broadband Probe

The low-frequency broadband probe is optimized for minimal acoustic ringing artifacts in the 12–40 MHz range. Minor residual acoustical ringing artifacts, however, do occur, but these can be minimized by optimizing the `rof2` parameter. The appropriate setting for the `rof2` parameter for the nucleus of interest can be determined in the following manner:

1. Determine the spectral parameters for the experiment of interest.
2. Remove the sample from the probe. Acquire a spectrum and display the FID.
3. The FID should not contain any signal. If a signal exists, determine its duration by positioning the left cursor at the first point in the FID and the right cursor just to the right of the residual signal. The screen displays the difference in the cursors in seconds (e.g., 2e-5).
4. Enter the duration value obtained above for `rof2` in microseconds (e.g., 20). Values of `rof2` less than 100 μs should minimize the residual signal.

Tuning Ranges for Broadband Probes

The following tables list the tuning ranges for broadband probes:

- For 300-MHz, 10-mm probes, see [Table 6](#).
- For 400-MH, 10-mm probes, see [Table 7](#).
- For 500-MHz 5-mm probes, see [Table 8](#)
- For 500-MHz, 10-mm probes, see [Table 9](#).
- For 600-MHz probes, see [Table 10](#).

Table 6. Tuning Ranges for 300-MHz, 10-mm Broadband Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Capacitor Stick (pF)</i>	<i>Range (MHz)</i>
none	73–135	40	38–40
3	66–73	50	36–38
11	60–66	60	33–36
19	50–60	70	31–33
27	45–50	80	30–31
36	40–45		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 7. Tuning Ranges for 400-MHz, 10-mm Broadband Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Capacitor Stick (pF)</i>	<i>Range (MHz)</i>
none	92–170	21	54–60
7	75–92	27	47–54
13	64–75	36	43–47
19	60–64	45	40–43

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 8. Tuning Ranges for 500-MHz, 5-mm Broadband Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (turns)</i>	<i>Range (MHz)^a</i>
none	105–190	13	190–203
3	95–105		
7	80–95		
13	68–80		
19	60–68		
23	56–60		
27	53–56		
33	50–53		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 9. Tuning Ranges for 500-MHz, 10-mm Broadband Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (turns)</i>	<i>Range (MHz)^a</i>
none	95–175	12	175–203
3	80–95		
11	64–80		
19	55–64		
26	50–55		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 10. Tuning Ranges for 600-MHz Broadband Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (turns)</i>	<i>Range (MHz)</i>
none	100–170	8	170–285
3	93–125		
7	76–90		
13	66–77		
21	57–64		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

3.4 Indirect Detection Probes

This section covers the following indirect detection and pulsed field gradient (PFG) indirect detection probes:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Range</i>	<i>Probe Part No.</i>
300	54	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-901981-00
300	89	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-901980-00
400	54	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-992804-00
400	89	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-993153-00
500	54	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-993079-00
500 PFG	54	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-993373-00
500 PFG	54	3	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	01-903441-00
600	54	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-993080-00
600 PFG	54	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-993374-00

This section is organized as follows:

- “Operational Considerations,” [this page](#)
- “Connecting to UNITY-Series or VXR” [page 75](#)
- “Connecting to MERCURY-Series, GEMINI 2000, or Gemini” [page 77](#)
- “Tuning the Probe” [page 78](#)
- “Returning to Normal Operation” [page 79](#)
- “Tuning Ranges for Indirect Probes” [page 79](#)

Operational Considerations

The $^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$ Indirect probe is specifically designed for demanding applications in high-field NMR, for example, heteronuclear 2D, 3D, and 4D experiments. The PFG version of the Indirect probe adds rapid recovery gradients, up to 30 G/cm along the Z axis, to the probe features. The PFG probe requires the Performa I or the Performa II Pulsed Field Gradient module (Part No. 00-901858-00 for Performa I or 00-993854-00 for Performa II). To achieve the level of performance required for these specialized experiments, the probe has been optimized in the following ways:

- *High-power handling* – The probe can typically handle the full pulse power (200–300 W) delivered by a spectrometer for up to a 180 pulse for nuclei between ^{15}N and ^{13}C . The probe is not optimized for ^{31}P and must be operated at ^{31}P with the reduced power (you can use $\text{tpwr} < 54$ as a guideline) and a 90 pulse width longer than 30 s. For longer pulses (for example, a composite 360 pulse), the power should be decreased (2 to 4 dB) to minimize the chance of arcing. The amount of power reduction required is best determined by experimentation. The probe is not usually damaged by short pulse arcing (under 80 s), but arcing during a long pulse can damage the probe.

- *Spin locking conditions –*

Sample Size (mm)	Channel	Gamma B_x	Duration	Approximate Power
5	^{13}C	9 kHz	30 ms	30 W
5	^{15}N	4 kHz	30 ms	30 W
3	^{13}C	11 kHz	30 ms	30 W
3	^{15}N	5 kHz	30 ms	30 W

- *Decoupling conditions –*

Channel	Channel	Gamma B_x	Duration	Approximate Power
5	^{13}C	7 kHz	150 ms	15 W
5	^{15}N	2 kHz	150 ms	7.5 W
3	^{13}C	9 kHz	150 ms	15 W
3	^{15}N	3 kHz	150 ms	7.5 W

- *Tuning* – Proper tuning is vital to short pulse width performance. (Always tune the X channel with no filter in line.)
- *Tuning range* – The broadband channel (labeled X CHANNEL on the probe) can tune to nuclei between ^{15}N and ^{31}P . A capacitor stick is provided to tune to ^{15}N and an inductor stick to tune to ^{31}P . The proton channel tunes to ^1H , except for 300-MHz probes. When turning the X channel on 300-MHz PFG Indirect probes, the familiar dip behavior for minimum reflected power may not be achievable on the tune meter for ^{15}N and ^{31}P . For these cases, tune the probe to the lowest meter value without forcing the matching capacitor (smooth knob) beyond its range. Adjustment to this meter value provides the maximum rated rf performance at these frequencies.

CAUTION: The Indirect probe can be damaged by high power. Be extremely careful when applying spin-locks or high-power decoupling to the probe. Also, arcing during a long pulse can damage the probe.

Connecting to UNITY-Series or VXR

To connect an Indirect or PFG Indirect probe to a ^{UNITY}INOVA, UNITYplus, UNITY, or VXR system:

1. If the probe mounting plate has not been adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#).
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided.
3. In the lock channel line, install the appropriate ^2H bandpass filter listed in the following table. Expect the lock phase to change upon addition of the filter. Note that

the ^2H bandpass filter can be left in the system at all times; however, it causes about 20% loss in lock sensitivity.

<i>Magnet (MHz)</i>	<i>^2H Bandpass Filter</i>
300	BE46-4-6BB
400	BE61-10-8BB
500	BE77-3.8-8BB
600	BE95-12-8BB

Filter model numbers describe the filter's function. For example, the 500-MHz bandpass filter model number is BE77-3.8-8BB, where the 77 indicates 77 MHz and "B" indicates bandpass—an "L" instead would indicate low-pass.

4. Connect the low-band decoupler cable according to the appropriate procedure below. Choose the appropriate bandpass filters from the following table:

<i>Magnet (MHz)</i>	<i>^{13}C Bandpass Filter</i>	<i>^{15}N Bandpass filter</i>
300	BE75-15-8BB	BE30.4-7.6-9BB
400	BE109-22-8BB	BE40-14-9BB
500	BE135-35-8BB	BE53-15-8BB
600	BE154-40-8BB	BE61-10-8BB

- For 300-MHz systems, connect a cable from the magnet leg B-Band Probe port ($^{\text{UNITY}}$ INOVA or UNITYplus), B-Band port (UNITY), or the Probe NORMAL port (VXR) to the appropriate bandpass filter. Then, attach a cable from the bandpass filter to the port labeled X CHANNEL.
 - For 400-MHz systems, connect a cable from the B Band port, or the Probe NORMAL port on the magnet leg, to the appropriate bandpass filter. Then attach a cable from the filter to the probe port labeled X CHANNEL.
 - For 500-MHz systems with a $^1\text{H}/^{19}\text{F}$ OBS preamplifier, connect a cable from the Decplr or NORMAL port to the appropriate bandpass filter. Then attach a cable from the filter to the probe port labeled X CHANNEL.
 - For 500-MHz systems with a dual preamplifier, connect a cable from the B Band CM port to the appropriate bandpass filter. Then connect a cable from the filter to the probe port labeled X CHANNEL.
 - For 600-MHz systems, connect the appropriate bandpass filter to the cable labeled "Indirect Detection" or "L Band Decpl." Then connect a cable from the filter to the probe port labeled X CHANNEL.
5. Connect the ^1H channel according to the appropriate step below:
 - For 300-MHz systems, connect a ^1H high-pass filter to the magnet leg H-Band Probe port ($^{\text{UNITY}}$ INOVA or UNITYplus), to the H-Band port (UNITY), or to the ^1H /OBS/DEC port (VXR). Then connect a cable from the filter to the PROTON port on the probe.
 - For 400-MHz systems, connect a ^1H high-pass filter to the H Band port or to the ^1H /OBS/DEC port. Then connect a cable from the filter to the PROTON port on the probe.
 - For 500-MHz systems with the $^1\text{H}/^{19}\text{F}$ OBS preamplifier, connect a ^1H high-pass filter to the PROBE port (J5301) on the preamplifier. Then connect the a cable from the filter to the PROTON port on the probe.

- For 500- and 600-MHz probes with the dual preamplifier, connect a ^1H high-pass filter to the H BAND CM or the PROBE port (J5301) on the preamplifier. Then connect a cable from the filter to the PROTON port on the probe.
6. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.
 7. Connect the nitrogen VT gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe. **Figure 3** shows a pneumatics connection diagram.
 8. Connect the body-cooling air hose to the hose barb protruding from the bottom of the probe. **Figure 3** shows a pneumatics connection diagram.

Connecting to **MERCURY-Series, GEMINI 2000, or Gemini**

The Indirect detection probe is physically installed with two mounting screws. Connecting the cables, however, is different than that of the $^1\text{H}/^{19}\text{F}$ probe.

Connecting the Cables for High-Band (^1H) Operation

1. In the lock channel line, install the ^2H bandpass filter; expect the lock phase to change upon addition of the filter. (The ^2H bandpass filter *can* be left in the system at all times; it causes, however, about 20 percent loss in lock sensitivity.)
2. The observe cable now carries X nucleus signals. Accordingly, attach the observe cable to the ^{13}C connector (J5302), insert an X-nucleus bandpass filter to the other end, and then connect the filter to the probe connector marked {x} DECOUPLE.
3. Install the appropriate quarter-wavelength cable on the broadband preamplifier. Connect the proper cable for the observe frequency, as indicated in the following table, to J5304 and J5305 on the left magnet leg.

Frequency (MHz)	Cable (Color)
44–178	Yellow
37–118	Black
11–45	Blue

4. Connect the probe connector of the proton preamplifier to a ^1H high-pass filter and connect the filter to the connector of the probe marked 1H OBSERVE.
5. If the VT option is present, connect the cable from VT controller to the 9-pin connector (VT Control) on the probe, and connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe.
6. Connect the body-cooling air hose to the hose barb on the bottom of the probe and adjust the air flow to optimize sample insertion, ejection, and spinning (refer to **Section 2.1, “Adjusting Spinning and Air Pressure,”** on page 24).

Connecting the Cables for Low-Band (Broadband) Operation

The indirect detection probe is also capable of being used as a conventional broadband probe with X-nucleus observe and ^1H decoupling. Connecting the cable for low band should be the same as the high-band operation cable connections described above—the observe signal should be connected to the probe connector marked {x} DECOUPLE and the decoupler signal should be connected to the probe connector marked 1H OBSERVE.

Tuning the Probe

The Indirect Detection probe performs best when the sample to be analyzed is placed in the probe during the tuning operation. For best probe performance, follow the ^1H channel and X channel tuning procedures below every time you change samples.

The Indirect probe, pictured in Figure 30, incorporates a numerical counter system and probe label on the broadband channel that corresponds to the tuning of the nuclei to be decoupled/observed. An example of the probe label is shown in Figure 31. Counter values are established for each probe, without a sample. The knurled tune knob changes the values on the numerical counter, while the smooth knob operates the matching capacitor. The lower the frequency of the nucleus, the more the smooth match knob needs to be turned clockwise.

Follow the appropriate tuning procedures for your system:

- UNITYINOVA, UNITYplus – See Section 2.3, “Tuning Probes with UNITYINOVA or UNITYplus,” on page 33.
- GEMINI 2000 – See Section 2.4, “Tuning Probes with MERCURY-Series or GEMINI 2000 Systems,” on page 37.
- UNITY, VXR – See Section 2.5, “Tuning Probes with UNITY or VXR,” on page 40.
- Gemini – See Section 2.6, “Tuning Probes with Gemini Systems,” on page 43.

CAUTION: Do not force the tuning capacitors to turn past the end of their ranges. The end of the range is indicated by an increase in torque and not necessarily a stop to the rotation. Forced turning can cause serious mechanical damage to the probe, requiring factory repair.

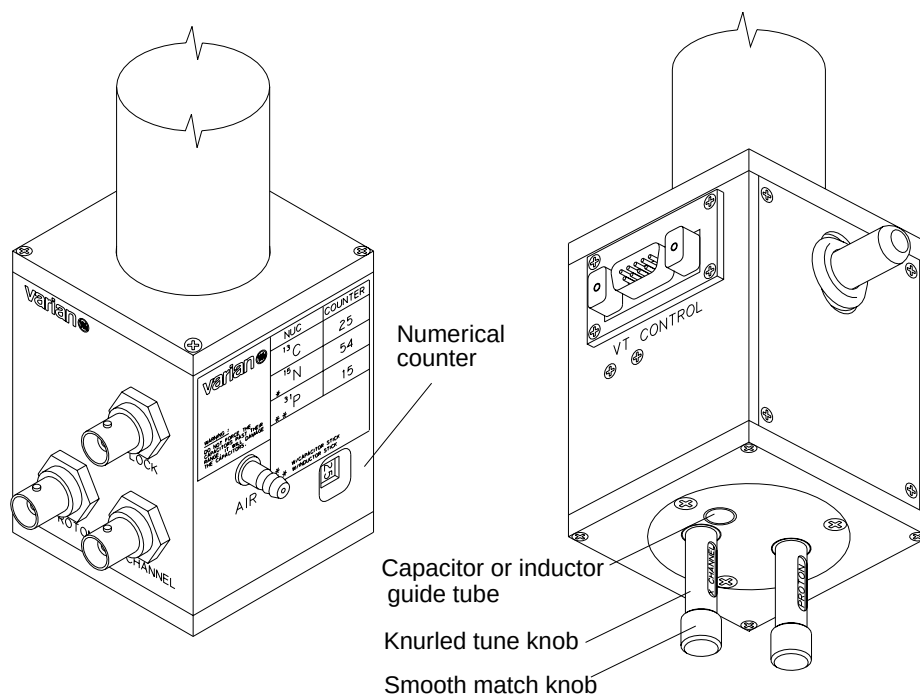


Figure 30. Indirect Detection Probe Tuning Controls

Tuning the ^1H Channel

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the red PROTON inset label turns the tuning capacitors, while the smooth lower knob turns the match capacitor (see [Figure 30](#)).

varian CPA 500 ID/PFG 50 – 202 MHz 0099337301 WARNING: DO NOT FORCE THE CAPACITORS PAST THEIR RANGE. IT WILL DAMAGE THE CAPACITORS.	NUC	COUNTER
	^{13}C	17
	* ^{31}P	26
	** ^{15}N	60
	^{29}Si	59
* 7T INDUCTOR STICK ** 30PF CAPACITOR STICK		

Tuning the X Channel

Turn the knurled X CHANNEL knob, shown in [Figure 30](#), to a numerical counter value that corresponds to the nucleus to be observed. The label on your probe, pictured in [Figure 31](#), shows four popular nuclei. Then, to fine tune, alternately adjust the smooth and knurled X CHANNEL match and tune knobs to find the minimum reflected power on the tune meter.

Figure 31. Indirect Detection Probe Label with Example Counter Values

When turning the X channel on 300-MHz PFG Indirect probes, the familiar dip behavior for minimum reflected power may not be achievable on the tune meter for ^{15}N and ^{31}P . For these cases, tune the probe to the lowest meter value without forcing the matching capacitor (smooth knob) beyond its range. Adjustment to this meter value provides the maximum rated rf performance at these frequencies.

Returning to Normal Operation

After tuning the probe, enter `dm='n' su` to make sure the decoupler is not inadvertently left on. Check ^1H channel tuning after X-channel tuning as well as after a sample change.

Tuning Ranges for Indirect Probes

This section provides tables listing tuning ranges for Indirect probes. Use these tables to choose the capacitor or inductor stick (note that some sticks are optional equipment):

- 300-MHz probes, see [Table 11](#).
- 400-MHz probes, see [Table 12](#).
- 500-MHz, see [Table 13](#).
- 600-MHz, see [Table 14](#).

Table 11. Tuning Ranges for 300-MHz Indirect Detection Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (Turns)</i>	<i>Range (MHz)</i>
none	60–78 (¹³ C, ²⁹ Si)	12	95–128 (³¹ P)
3	51.9–67		
10	44.5–52		
14.3	42.8–48		
21	37.8–41		
26	35–37.8		
30	33.3–35		
33	32.8–34.8		
43	30.2–32		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 12. Tuning Ranges for 400-MHz Indirect Detection Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (Turns)</i>	<i>Range (MHz)</i>
none	75–102 (¹³ C)	7	110–190 (³¹ P)
9	60–74		
14	54–61		
17	54–56		
23	50–55		
27	47–54		
30	45–49		
36	42–46		
43	40.4–42.4 (¹⁵ N)		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 13. Tuning Ranges for 500-MHz Indirect Detection Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (turns)</i>	<i>Range (MHz)</i>
none	78–133 (¹³ C)	7	130–210 (³¹ P)
9	70–87	9	125–185
10	65–79		
14	60–70		
17	55–62		
23	53–57		
27	47–55 (¹⁵ N)		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 14. Tuning Ranges for 600-MHz Indirect Detection Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (turns)</i>	<i>Range (MHz)^a</i>
none	100–153 (¹³ C)	5	190–245 (³¹ P)
5	80–104	7	155–190
10	70–83		
14	63–73		
19	57–63 (¹⁵ N)		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

3.5 Switchable Probes

This section covers the following Switchable probes:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Range</i>	<i>Probe Part No.</i>
200	51	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	01-902651-00
200	54	5	$^1\text{H}-^{19}\text{F}/^{15}\text{N}-^{31}\text{P}$	00-969835-00
200	89	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	01-902654-00
200	54	5	$^1\text{H}/^{13}\text{C}$	01-904511-00
200	51	5	$^1\text{H}/^{13}\text{C}$	01-904515-00
300	51	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	01-902632-00
300	54	5	$^1\text{H}/^{13}\text{C}$	01-904510-00
300	51	5	$^1\text{H}/^{13}\text{C}$	01-904514-00
300	89	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	01-902635-00
200	89	5	$^1\text{H}-^{19}\text{F}/^{15}\text{N}-^{31}\text{P}$	00-969836-00
400	51	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	01-902638-00
400	89	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	01-902643-00
500	51	5	$^1\text{H}\{^{15}\text{N}-^{31}\text{P}\}$	00-993375-00

This section is organized as follows:

- “Connecting to UNITY-Series or VXR,” [this page](#)
- “Connecting to MERCURY-Series, GEMINI 2000, or Gemini” [page 83](#)
- “Tuning the Probe” [page 84](#)
- “Returning to Normal Operation” [page 85](#)
- “Tuning Ranges for Switchable Probes” [page 85](#)

Connecting to UNITY-Series or VXR

To connect a switchable probe to a ^{UNITY}INOVA, UNITY*plus*, UNITY, or VXR system:

1. If the probe mounting plate is not adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#).
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided.
3. Connect the LK PROBE connector (P5205), which is inside the magnet leg on top of the lock preamplifier, to the 250-MHz low-pass filter mounted on the back of the magnet leg, and then connect the filter to the LOCK connector on the probe.
4. Connect the observe low band cable using the appropriate step below:
 - *200-, 300-, and 400-MHz systems using broadband preamplifier* – Connect an appropriate low-pass and notch filter in line to the B BAND or OBSERVE NORMAL port on the magnet-console interface (magnet leg). Connect a cable from the filters to the X CHANNEL port on the probe.
 - *500-MHz systems using dual preamplifier* – Connect an appropriate low-pass and notch filter in line to the PROBE port (J5311) on the broadband preamplifier, and then connect a cable from the filters to the X CHANNEL port on the probe.

Omit the notch filter when observing ^2H or other nuclei whose resonance frequency is close to 76 MHz.

5. Connect the decoupler cable using the appropriate step below:
 - *Systems using $^1\text{H}/^{19}\text{F}$ OBS preamplifier* – Connect a cable and a high-pass filter between the 1H/OBS/DEC port on the magnet-console interface (magnet leg) and the PROTON port on the probe.
 - *200-, 300-, and 400-MHz systems using broadband preamplifier* – Connect a cable and a high-pass filter between the H BAND or 1H/OBS/DEC port on the preamplifier and the PROTON port on the probe.
 - *500-MHz systems using dual preamplifier* – Connect a cable and a high-pass filter between the PROBE port (J5301) and the PROTON port on the probe.
6. Select an external quarter-wavelength cable to match the frequency of the observe nucleus. Attach this cable between the two BNC connectors labeled 1/4 WAVELENGTH on the preamplifier.
7. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.
8. Connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe. **Figure 3** shows a pneumatics connection diagram.
9. Connect the body-cooling air hose to the hose barb protruding from the bottom of the probe. **Figure 3** shows a pneumatics connection diagram.

Connecting to **MERCURY-Series** , **GEMINI 2000**, or **Gemini**

To connect a switchable probe to a **MERCURY-VX**, **MERCURY**, **GEMINI 2000**, or **Gemini** system:

1. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided.
2. Connect the LK PROBE connector (P5202) to a low-pass notch filter, and then connect the filter to the LOCK connector on the probe.
3. Connect a cable from the ^{13}C connector (J5302) on the left magnet leg to the OBS connector on the probe.
4. On broadband systems, in order to observe some nuclei, an appropriate quarter-wavelength cable must be installed on the broadband preamplifier. Connect the cable for the observe frequency, as indicated in the following table, to J5304 and J5305 on the left magnet leg.

<i>Frequency (MHz)</i>	<i>Cable (Color)</i>
44–178	Yellow
37–118	Black
11–45	Blue

5. Connect the decoupler cable (from the ^1H connector J5102) to the DEC connector on the probe. A ^1H high-pass filter must also be connected between ^1H connector (J5102) and the coax cable.

6. If the VT option is present, connect the cable from VT controller to the 9-pin connector (VT Control) on the probe, and connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe.
7. Connect the body-cooling air hose to the hose barb on the bottom of the probe and adjust the air flow to optimize sample insertion, ejection, and spinning (refer to [Section 2.1, “Adjusting Spinning and Air Pressure,”](#) on page 24).

Tuning the Probe

For best probe performance, place the sample to be analyzed in the probe during tuning and follow the ^1H channel and X channel tuning procedures every time you change samples.

The Switchable probe, shown in [Figure 32](#), incorporates a numerical counter system and probe label on the broadband channel that corresponds to the tuning of the nuclei to be decoupled/observed. An example of the probe label is shown in [Figure 33](#). The knurled tune knob changes the values on the numerical counter, while the smooth knob operates the matching capacitor. The lower the frequency of the nucleus, the more the smooth match knob needs to be turned clockwise.

CAUTION: Do not force the tuning capacitors to turn past the end of their ranges. The end of the range is indicated by an increase in torque and not necessarily a stop to the rotation. Forced turning can cause serious mechanical damage to the probe, requiring factory repair.

To tune the probe, refer to the tuning procedure for your system:

- UNITYINOVA, UNITYplus – See [Section 2.3, “Tuning Probes with UNITYINOVA or UNITYplus,”](#) on page 33.

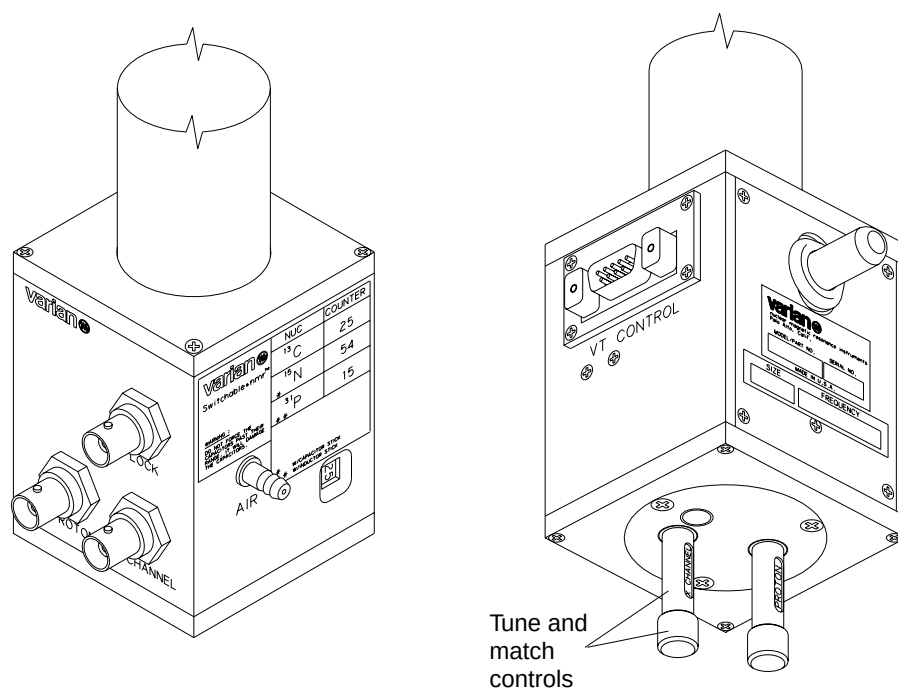


Figure 32. Switchable Probe Connectors and Tuning Controls

- *MERCURY-VX, MERCURY, GEMINI 2000* – See Section 2.4, “Tuning Probes with MERCURY-Series or GEMINI 2000 Systems,” on page 37.
- *UNITY, VXR* – See Section 2.5, “Tuning Probes with UNITY or VXR,” on page 40.
- *Gemini* – See Section 2.6, “Tuning Probes with Gemini Systems,” on page 43.

Tuning the ^1H Channel

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the red PROTON inset label turns the tuning capacitors, while the smooth lower knob turns the match capacitor (see Figure 32). When tuning to ^{19}F , adjust the knurled knob in the counter-clockwise direction and adjust the match capacitor.

Tuning the X Channel

Turn the knurled X CHANNEL knob, shown in Figure 32, to a numerical counter value that corresponds to the nucleus to be observed. The probe label (see Figure 33) shows four popular nuclei. To fine tune, alternately adjust the smooth and knurled X CHANNEL match and tune knobs to find the minimum reflected power on the tune meter.

varian  500 SW 50 – 203 MHz 0099337500 WARNING: DO NOT FORCE THE CAPACITORS PAST THEIR RANGE. IT WILL DAMAGE THE CAPACITORS.	NUC	COUNTER
	^{13}C	25
	* ^{31}P	15
	** ^{15}N	54
	^{29}Si	43
* 8T INDUCTOR STICK ** 21PF CAPACITOR STICK		

Figure 33. Switchable Probe Label Example

Returning to Normal Operation

After tuning the probe, enter `dm='n' su` to make sure the decoupler is not inadvertently left on. Check the ^1H channel tuning after X-channel tuning and after a sample change.

Tuning Ranges for Switchable Probes

The following tables list tuning ranges for switchable probes:

- 200-MHz probes, see Table 15.
- 300-MHz probes, see Table 16.
- 400-MHz probes, see Table 17.
- 500-MHz probes, see Table 18.

Table 15. Tuning Ranges for 200-MHz Switchable Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Capacitor Stick (pF)</i>	<i>Range (MHz)</i>
none	43–83	36	25–28
11	34–45	50	22–24
19	30–37	66	19.5–22
27	27–31		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 16. Tuning Ranges for 300-MHz Switchable Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Capacitor Stick (pF)</i>	<i>Range (MHz)</i>
none	55–125	30	32–36
11	42–57	36	30–33
23	35–42	43	28–31

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 17. Tuning Ranges for 400-MHz Switchable Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Capacitor Stick (pF)</i>	<i>Range (MHz)</i>
none	90–173	36	46–51
9	70–90	45	43–46
19	57–70	53	41–43
27	51–57	55	40–41

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

Table 18. Tuning Ranges for 500-MHz Switchable Probes

<i>Capacitor Stick (pF)</i>	<i>Range (MHz)¹</i>	<i>Inductor Stick (turns)</i>	<i>Range (MHz)</i>
none	85–137 (¹³ C)	9	135–212 (³¹ P)
9	66–82		
10	65–79		
14	60–70		
17	55–62		
23	53–57		
27	48–54 (¹⁵ N)		

1. Ranges are approximate and valid for empty probes only. Ranges are slightly lower with a sample in a probe.

3.6 Triple-Resonance Probes

This section covers the following Triple-Resonance PFG (Pulsed Field Gradient) Triple Resonance probes:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Range</i>	<i>Probe Part No.</i>
400	54	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	00-993206-00
400	89	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	00-993207-00
500	54	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	00-993077-00
500	54	5	$^1\text{H}\{^{13}\text{C}/^{31}\text{P}\}$	00-993150-00
500	54	5	$^{13}\text{C}\{^1\text{H}/^{15}\text{N}\}$	00-991100-00
500 PFG	54	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	00-992430-01
500 PFG	54	8	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	01-903028-00
500 XYZ PFG	54	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	01-901589-00
500 PFG	54	3	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	01-902867-00
600	54	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	00-993078-00
600	54	5	$^{13}\text{C}\{^1\text{H}/^{15}\text{N}\}$	00-991102-00
600 PFG	54	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	00-992910-00
600 PFG	54	5	$^1\text{H}\{^{13}\text{C}/^{31}\text{P}\}$	01-903768-00
600 PFG	54	8	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	01-902809-00
600 XYZ PFG	54	5	$^1\text{H}\{^{13}\text{C}/^{15}\text{N}\}$	01-901592-00

This section is organized as follows:

- “Operational Considerations,” [this page](#)
- “Connecting the Probe” [page 89](#)
- “Tuning the Probe” [page 90](#)

Operational Considerations

The Triple-Resonance probe is specifically designed for demanding applications in biomedical NMR, such as, heteronuclear 2D, 3D, and 4D Triple resonance experiments on isotopically enriched proteins. To achieve the required level of performance for these specialized experiments, the probe has been optimized in the following ways that should be kept in mind during its use.

The Pulsed Field Gradient version of the Triple Resonance probe adds rapid recovery gradients, up to 65 G/cm along the Z axis, to the probe features. The PFG probe requires the Pulsed Field Gradient Module (00-992915-00, 00-992916-00).

The XYZ PFG version of the Triple Resonance probe adds X (25 G/cm), Y (25 G/cm), and Z (65 G/cm) gradient capability to the probe features. The XYZ PFG probe requires the XYZ PFG module for UNITYINOVA (01-903977-00) or UNITYplus (01-903977-01).

- *High power handling* – The probe can typically handle full pulse power (200–300 W) for up to a 180 pulse.
- *Simultaneous pulse conditions* – Find the maximum power (P_{max}) that the carbon channel can take without arcing. Then, for simultaneous carbon and nitrogen pulses, apply the following derating relation:

$$P_{^{13}\text{C}} + P_{^{15}\text{N}} + 2\sqrt{P_{^{15}\text{N}}P_{^{13}\text{C}}} \leq P_{\text{max}}$$

This derating ensures trouble-free probe operation. However, provided that the *pulses are less than 100 s long* slightly more power (to near the point of arcing) can be applied without damaging the probe.

CAUTION: To prevent damage to the probe, be extremely careful when applying spin-locks or high-power decoupling to the probe.

- *Spin locking conditions –*

Channel	Gamma B_x	Duration	Approximate Power
^{13}C	7 kHz	30 ms	30 W
^{15}N	2 kHz	30 ms	30 W
^{31}P	2 kHz	30 ms	30 W

- *Decoupling conditions –*

Channel	Gamma B_x	Duration	Approximate Power
^{13}C	5 kHz	150 ms	15 W
^{15}N	1 kHz	150 ms	7.5 W
^{31}P	1 kHz	150 ms	1.5 W

- *Non-spinning operation* – The probe is intended for cancellation experiments that are typically performed non-spinning. Non-spinning performance has therefore been optimized relative to spinning performance.
- *Tuning* – Proper tuning is vital to short pulse width performance. *Always tune the probe with no filter in line.* Once tuned, attach the required filter using the shortest cables available.
- *3-mm tuning range, sample volume, and air flow adjustments* – The 3-mm probe tunes to the specified nucleus on each channel with up to 1 M sample loading. None of the four channels is broadband tunable. The recommended minimum sample height for 3-mm sample tubes (Wilmad 327-PP or equivalent) is approximately 33 mm (150 μl). Due to the small sample dimensions, VT air flow should be reduced to about 5–9 lpm to avoid sample lifting.
- *5-mm tuning range* – The 5-mm probe tunes to the specified nucleus on each channel with as much as 1 M sample loading. None of the four channels is broadband tunable; however, the proton channel tunes to ^{19}F and the carbon channel has a 10-MHz range centered approximately on ^{13}C frequency.
- *8-mm tuning range and sample geometry* – To ensure the highest proton sensitivity at lower salt concentrations, the 8-mm probe is designed to tune to 250 mM of salt. Using smaller diameter sample tubes (i.e., 6-mm or 7-mm) can help optimize sample geometry and salt tolerance. For ionic samples in an 8-mm tube, the pw90 might increase by as much as 1.5 times for a 100 mM solution and 1.8 times for a 250 mM solution. For concentrations higher than 250 mM, using smaller diameter sample tubes or using 5-mm probes might be more efficient. Restricting the sample height by using Shigemi sample tubes also reduces sample loading and thus the degradation of pulse width. The optimum sample height for the 8-mm Triple Resonance PFG probe is 50 mm.

- *8-mm air flow* – For the 8-mm probe, we recommend using 10 to 15 lpm if VT air flow and 5 to 10 lpm of body cooling air. For longer-duration decoupling, you can increase the body cooling air.

Connecting the Probe

The Triple-Resonance probe is installed with two mounting screws, the same as other probes. To produce simultaneous high-power ^{13}C and ^{15}N pulses (or decoupling) at the probe, the spectrometer must be equipped with a third channel accessory. The third channel, because of the absence of routing relays, typically delivers higher power than the main decoupler and should be connected to the NITROGEN port of the probe.

1. In the lock channel line, install the appropriate ^2H bandpass filter listed in the following table. Expect the lock phase to change when the filter is added. Note that the ^2H bandpass filter can be left in the system at all times; however, it causes about 20% loss in lock sensitivity.

<i>Magnet (MHz)</i>	<i>^2H Bandpass Filter</i>
400	BE61-10-8BB
500	BE77-3.8-8BB
600	BE95-12-8BB

Filter model numbers describe the filter's function. For example, the 500-MHz bandpass filter model number is BE77-3.8-8BB, where the 77 indicates 77 MHz and "B" indicates bandpass; an "L" instead would indicate low-pass.

2. Connect the low band decoupler cable according to the appropriate procedure below. Choose the appropriate bandpass filters from the following table:

<i>Magnet (MHz)</i>	<i>^{13}C Bandpass Filter</i>	<i>^{15}N Bandpass filter</i>
400	BE109-22-8BB	BE40-14-9BB
500	BE135-35-8BB	BE53-15-8BB
600	BE154-40-8BB	BE61-10-8BB

- *400-MHz systems* – Connect a cable from the B Band port, or the Probe NORMAL port on the magnet leg, to the appropriate bandpass filter. Then attach a cable from the filter to the probe port labeled CARBON.
 - *500-MHz systems with $^1\text{H}/^{19}\text{F}$ OBS preamplifier* – Connect a cable from the Decpl or NORMAL port to the appropriate bandpass filter. Then attach a cable from the filter to the probe port labeled CARBON.
 - *500-MHz systems with dual preamplifier* – Connect a cable from the B Band CM port to the appropriate bandpass filter. Then connect a cable from the filter to the probe port labeled CARBON.
 - *600-MHz systems* – Connect the appropriate bandpass filter to the cable labeled "Indirect Detection" or "L Band Decpl." Then connect a cable from the filter to the probe port labeled CARBON
3. Attach the third channel cable to a ^{15}N bandpass filter (see the table above for filter model number) and then to the probe connector marked NITROGEN.
 4. Connect the ^1H channel according to the appropriate step below:

- *400-MHz systems* – Connect a ^1H high-pass filter to the H Band port or 1H/OBD/DEC port. Then connect a cable from the filter to the PROTON port on the probe.
 - *500-MHz systems with $^1\text{H}/^{19}\text{F}$ OBS preamplifier* – Connect a ^1H high-pass filter to the PROBE port (J5301) on the preamplifier. Then connect the a cable from the filter to the PROTON port on the probe.
 - *500- and 600-MHz probes with dual preamplifier* – Connect a ^1H high-pass filter to the H BAND CM or the PROBE port (J5301) on the preamplifier. Then connect a cable from the filter to the PROTON port on the probe.
5. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.
 6. Connect the VT nitrogen gas hose to the stub tee on the glass dewar projecting to the side at the base of the probe. **Figure 3** shows a pneumatics connection diagram.
 7. Connect the body-cooling air hose to the hose barb protruding from the bottom of the probe. **Figure 3** shows a pneumatics connection diagram.

Tuning the Probe

CAUTION: Do not force the tuning capacitors to turn past the end of their ranges. The end of the tuning range is indicated by an increase in torque and not necessarily a stop to the rotation. Forced tuning can cause serious mechanical damage to the probe, requiring factory repair.

The Triple-Resonance probe performs best when the sample to be analyzed is placed in the probe during the tuning operation and tuning procedures are repeated every time you change samples. Because the probe channels are slightly interactive, repeat the ^1H , ^{13}C , and ^{15}N tuning procedures, checking to see if each channel shows zero reflected power on the tune meter. Make minor adjustments to the probe as necessary.

Refer to the section covering the tuning procedures for your system:

- *UNITYINOVA, UNITYplus* – See **Section 2.3, “Tuning Probes with UNITYINOVA or UNITYplus,”** on page 33.
- *UNITY, VXR* – See **Section 2.5, “Tuning Probes with UNITY or VXR,”** on page 40.

Tuning the ^1H Channel

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the red PROTON inset label turns the tuning capacitors and the smooth lower knob turns the match capacitor (see [Figure 34](#)). Turning the knurled knob clockwise (as viewed from the bottom of the probe) decreases tune capacitance, while turning the lower (smooth) knob clockwise increases the match capacitance.

Tuning the ^{13}C Channel

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the white CARBON inset label turns the tuning capacitors and the smooth lower knob turns the match capacitor (see [Figure 34](#)). Turning the knurled knob clockwise (as viewed from the bottom of the probe) decreases tune capacitance, while turning the lower (smooth) knob clockwise increases the match capacitance.

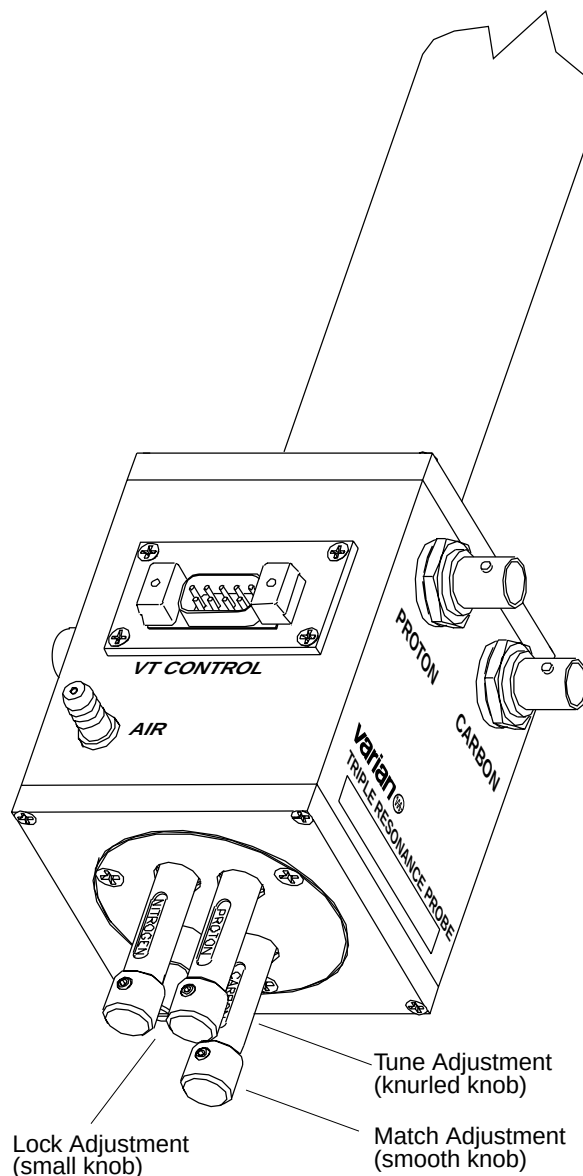


Figure 34. Triple-Resonance Probe Tuning Knobs

Tuning the ^{15}N Channel

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the yellow NITROGEN inset label turns the tuning capacitors and the smooth lower knob turns the match capacitor (see [Figure 34](#)). Turning the knurled knob clockwise (as viewed from the bottom of the probe) increases tune capacitance, while turning the lower (smooth) knob clockwise increases the match capacitance.

Chapter 4. Nano Probes

Sections in this chapter:

- 4.1 “Using the Bayonet Probe Flange” page 94
- 4.2 “Connecting the Proton Nano Probe” page 95
- 4.3 “Connecting the Carbon Nano Probe” page 96
- 4.4 “Connecting the Nano Probe to the Pneumatics/Tachometer Box” page 97
- 4.5 “Preparing Samples” page 99
- 4.6 “Resin Bead Samples” page 101
- 4.7 “Sample Spinning” page 101
- 4.8 “Installing Nano Sample Plugs Using the Plug Tool” page 103
- 4.9 “Probe Tuning” page 103
- 4.10 “Returning to Normal Operation” page 105
- 4.11 “Shimming the Nano Probe” page 105
- 4.12 “Performing an Experiment” page 106
- 4.13 “Changing Samples or Solvents” page 107
- 4.14 “Choosing a Sample Cell Filling Device” page 107
- 4.15 “Using the Sample Cleaning Holder” page 107

Nano probes provide small sample volumes and magic-angle spinning for high-resolution spectra of heterogeneous samples. Nano probes are also optimized for detection of samples where sample amount is limited.

This chapter covers the proton and carbon Nano probes listed below:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Probe Type</i>	<i>Sample Size (mm)</i>	<i>Probe Part No.</i>
300	54	300 NB ^1H Nano	4	00-993661-00
300	89	300 WB ^1H Nano	4	01-903660-00
400	54	400 NB ^1H Nano	4	00-993474-00
400	89	400 WB ^1H Nano	4	00-993474-01
500	51	500 ^1H Nano	4	01-903663-00
600	51	600 ^1H Nano	4	01-903664-00
400	54	400 NB $^{13}\text{C}[^1\text{H}]$ Nano	4	01-903667-00
400	89	400 WB $^{13}\text{C}[^1\text{H}]$ Nano	4	01-903668-00
500	51	500 $^{13}\text{C}[^1\text{H}]$ Nano	4	01-903669-00
600	51	600 $^{13}\text{C}[^1\text{H}]$ Nano	4	01-903670-00

4.1 Using the Bayonet Probe Flange

Some Nano probes are equipped with a bayonet probe flange, shown in [Figure 35](#), that allows quick and easy sample changing. The bayonet flange attaches the probe to the magnet—a plastic ring is clamped to the probe, and a metal base plate is attached to the magnet.

Inserting the Probe

A Nano probe with a bayonet flange is installed as follows:

1. Make sure the plastic ring is attached to the probe and the metal base plate is attached to the magnet.
2. Insert the probe upward into the magnet, lining up the tapered cutouts in the plastic ring with the three locating pins.
3. After the plastic ring is fully seated into the base plate, turn the probe counterclockwise (as viewed from the top of the probe) until you hear a click.

The spring-loaded plunger clicks when the probe locks into place.

If you don't hear the click, make sure the probe is locked into place. Sometimes the spring plunger stays in the out position. Ideally, the plunger should click into the hole in the plastic ring for correct operation.

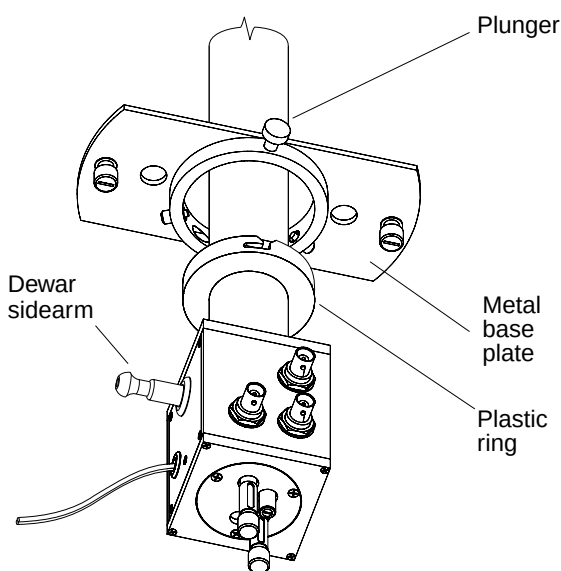


Figure 35. Nano Probe with Bayonet Probe Flange

Removing the Probe

A Nano probe with a bayonet flange is removed as follows:

CAUTION: The dewar sidearm is fragile and can break if the probe is twisted and the VT gas connection puts pressure on the sidearm. When removing the probe with the VT gas connection in place, do not put pressure on the dewar sidearm.

1. Pull out the spring-loaded plunger.
2. Rotate the probe 10 degrees clockwise (as viewed from the top of the probe).
3. Pull the probe downward slowly until the probe is clear of the magnet.

Changing Samples

Samples can be exchanged using the bayonet probe flange even with cables and gas lines still attached to the Nano probe.

4.2 Connecting the Proton Nano Probe

Connections between a proton Nano probe and a system differ depending on the system.

Connecting to UNITY-Series or VXR

To connect a proton Nano probe to a ^{UNITY}INOVA, UNITY*plus*, UNITY, or VXR system.

1. If the probe mounting flange is not adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#).
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided. If the probe uses the bayonet flange, refer to the section [Section 4.1, “Using the Bayonet Probe Flange,” on page 94](#).
3. Connect the LK PROBE connector (J5205), which is inside the magnet leg on top of the lock preamplifier, to a 250-MHz low-pass filter mounted on the magnet leg, and then connect a cable from the filter to the LOCK connector on the probe.
4. Connect the observe cable according the appropriate step below:
 - For the dual preamplifier, connect a proton frequency high-pass filter to the PROBE connector (J5301) on the preamplifier box. Then, connect a cable from the filter to the PROTON connector on the probe.
 - For the ¹H/¹⁹F OBS preamplifier, connect a proton frequency high-pass filter to the 1H/OBS/DEC connector on the preamplifier. Then, connect a cable between the filter and the PROTON connector on the probe.
5. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.

Connecting to MERCURY-Series, GEMINI 2000, or Gemini

To connect a proton Nano probe to a MERCURY-VX, MERCURY, GEMINI 2000, or Gemini system:

1. If the probe mounting flange is not adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#).
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided. If the probe uses the bayonet flange, refer to the section [Section 4.1, “Using the Bayonet Probe Flange,” on page 94](#).
3. Connect a lowpass notch filter to the ²H connector (J5202) on the left magnet leg. Connect a cable from the filter to the LOCK connector on the probe.
4. Connect a proton-frequency high-pass filter to the ¹H connector (J5402) on the left magnet leg. Then connect the cable from the high-pass filter to the PROTON connector on the probe.
5. If the VT option is present, connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.
6. Connect the body-cooling air hose to the hose barb on the bottom of the probe and adjust the air flow to optimize sample insertion, ejection, and spinning (refer to [Section 2.1, “Adjusting Spinning and Air Pressure,” on page 24](#)).

4.3 Connecting the Carbon Nano Probe

Connections between a carbon Nano probe and a system differ depending on the system.

Connecting to UNITY-Series or VXR

To connect a carbon Nano probe to a ^{UNITY}INOVA, UNITY*plus*, UNITY, or VXR system:

1. If the probe mounting flange is not adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,”](#) on page 18.
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided. If the probe uses the bayonet flange, refer to the section [Section 4.1, “Using the Bayonet Probe Flange,”](#) on page 94.
3. In the lock channel line, install the appropriate ²H bandpass filter listed in the following table. Expect the lock phase to change upon addition of the filter. Note that the ²H bandpass filter can be left in the system at all times; however, it causes about 20% loss in lock sensitivity.

<i>Magnet (MHz)</i>	<i>²H Bandpass Filter</i>
300	BE46-4-6BB
400	BE61-10-8BB
500	BE77-3.8-8BB
600	BE95-12-8BB

Filter model numbers describe the filter’s function. For example, the 500-MHz bandpass filter model number is BE77-3.8-8BB, where the 77 indicates 77 MHz and “B” indicates bandpass—an “L” instead would indicate low-pass.

4. Connect the observe channel according to the appropriate step below:
 - For the broadband preamplifier, connect the low-pass filter to the B BAND connector or the OBSERVE NORMAL on the magnet-console interface (magnet leg). Connect a cable from the low-pass filter to the OBS connector on the probe.
 - For the dual preamplifier, connect a low-pass filter to J5311 on the preamplifier. Connect a cable between the low-pass filter and the B BAND connector (K5008 NC). Connect a cable from the B BAND connector (K5008 CM) to the OBS connector on the probe.
5. Connect the decoupler channel according to the appropriate step below:
 - For the broadband preamplifier, connect a band-pass filter to the 1H/OBS/DEC or HB OBS/DCPL connector on the magnet-console interface (magnet leg). Connect a cable from the high-pass filter to the DEC connector on the probe.
 - For the dual preamplifier, connect a cable between the band-pass filter located on the side of the preamplifier assembly and the PROTON connector on the probe.
6. Select an external quarter-wavelength cable to match the frequency of the observe nucleus. Attach this cable between the two BNC connectors labeled 1/4 WAVELENGTH on the preamplifier.
7. Connect the cable from the variable temperature controller (VTC) to the 9-pin connector (VT Control) on the probe.

8. Connect the nitrogen VT gas line to the ball end of the glass dewar projecting to the side at the base of the probe. Refer to section [Section 4.4, “Connecting the Nano Probe to the Pneumatics/Tachometer Box,”](#) on page 97. See [Figure 3](#) for a general connection diagram.
9. Connect the body-cooling air hose to the hose barb protruding from the bottom of the probe. [Figure 3](#) shows a pneumatics connection diagram.

Connecting to *MERCURY-Series*, *GEMINI 2000*, or Gemini

To connect a carbon Nano probe to a *MERCURY-VX*, *MERCURY*, *GEMINI 2000*, or Gemini system:

1. If the probe mounting flange is not adjusted yet for the magnet, adjust the plate as described in [Section 1.3, “Installing the Probe and Upper Barrel,”](#) on page 18.
2. Insert the probe into the bottom of the magnet dewar. Secure the probe with the two thumbscrews provided. If the probe uses the bayonet flange, refer to the section [Section 4.1, “Using the Bayonet Probe Flange,”](#) on page 94.
3. Connect a lowpass filter to the ^{13}C connector (J5302) on the magnet leg. Connect a cable from the filter to the OBS connector on the probe.
4. Connect a lowpass notch filter to the ^2H connector (J5202). Connect a cable from the filter to the LOCK connector on the probe.
5. Connect the appropriate quarter-wavelength cable on the broadband preamplifier. Connect the proper cable for the observe frequency, as indicated in the following table, to J5304 and J5305 on the left magnet leg.

<i>Frequency (MHz)</i>	<i>Cable (Color)</i>
44–178	Yellow
37–118	Black
11–45	Blue

6. Connect the decoupler/proton observe cable from the ^1H connector (J5102) to the DEC connector on the probe. Also connect a ^1H high-pass filter between the ^1H connector (J5102) and the coax cable.
7. Connect the nitrogen VT gas line to the ball end of the glass dewar projecting to the side at the base of the probe. Refer to section [Section 4.4, “Connecting the Nano Probe to the Pneumatics/Tachometer Box,”](#) on page 97. See [Figure 3](#) for a general connection diagram.
8. Connect the cooling gas hose at the bottom of the probe housing to the cooling gas supply hose through the double-barbed connector supplied. This connection provides cooling for the probe body and allows adjustment of temperature over a small range near room temperature if the supply gas temperature can be varied.

4.4 Connecting the Nano Probe to the Pneumatics/Tachometer Box

To connect the Nano probe to the Pneumatics/Tachometer Box:

CAUTION: The stator, coil and other elements in the Nano probe can be damaged by contaminants in the gas supplied to the probe. The gas supplied to the probe should be dry, clean, and free of any contaminants whatsoever. If the probe is to be used at higher than room temperature (staying within the specification of the probe, of course) the use of standard air, containing oxygen, could result in oxidation of the coil structure. If the probe is to be used at lower than room temperature (again, staying within the specification of the probe), standard air, which often contains moisture, may allow ice buildup within the probe, which may affect circuit performance and spinning. In addition, often “house air”, which is pressurized using a compressor, is notoriously contaminated with oil and metal particles from the compressor. Contaminants such as these will cause all kinds of problems with any NMR probe, and especially with the Nano probe. *Dry nitrogen is recommended.*

1. Attach a ball joint connector (Part No. 00-993378-00) to one of the output lines of the Pneumatics/Tachometer Box using the quick disconnect. Refer to [Figure 36](#) as a reference. The Nano probe requires only one of the two output lines—either one works, but use the appropriate regulator and gauge to operate.
2. Attach the female ball end of the ball joint connector to the male ball end of the probe dewar. Tighten the connection by using a screw-tightening pinch clamp (Part No. 87-998700-00). Use only enough force to hold the two ends firmly together, since excessive force can damage these parts.

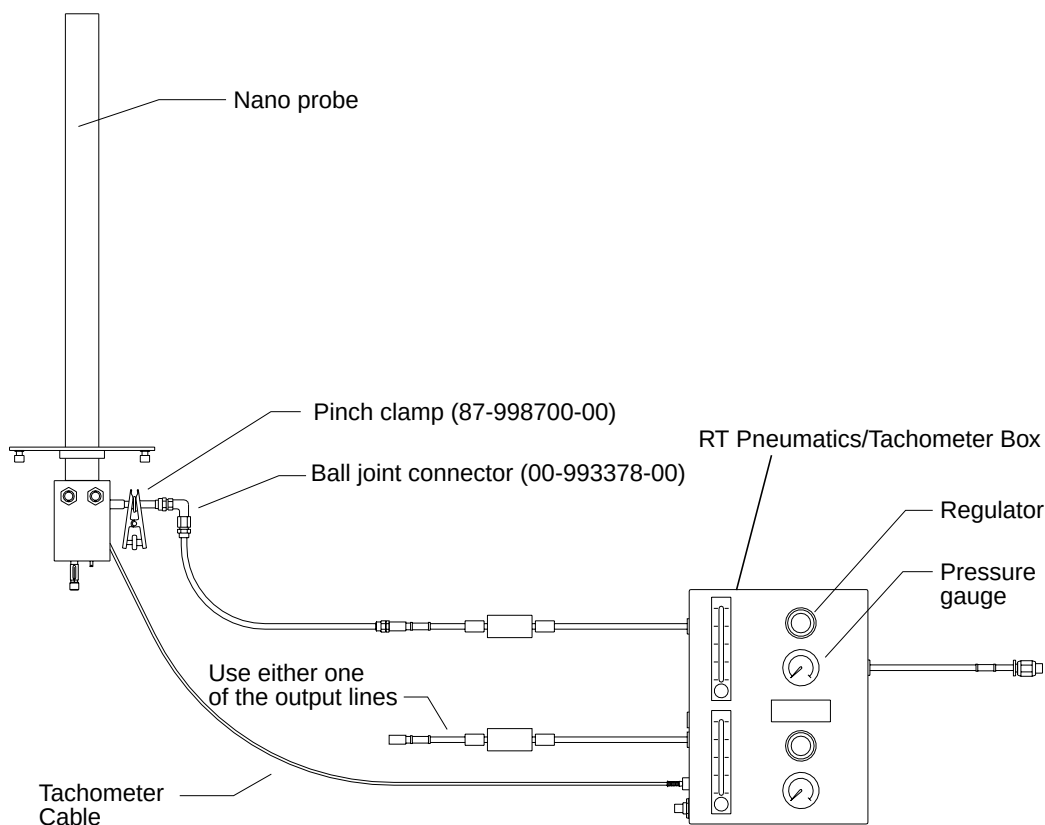


Figure 36. Nano Probe Pneumatics/Tachometer Connections

Note that when in use, the probe is pressurized from about 5 psig to about 30 psig, so the pinch clamp must be tight enough to prevent leaking at the joint. To be absolutely safe when inserting and removing the probe from the magnet bore, disconnect the ball joint connector from the probe, since movement of the connector may torque the probe dewar and damage it. However, the ball joint connector can remain attached if care is exercised. Refer to the section, [Section 4.1, “Using the Bayonet Probe Flange,”](#) on page 94.

3. Handling the tachometer cable by the black connectors, plug the tachometer cable into the Pneumatics/Tachometer Box. The cable ends must seat correctly to guarantee accurate spin rates.
4. Connect the body-cooling gas hose to the hose barb protruding from the side of the probe. [Figure 3](#) shows a pneumatics connection diagram.

4.5 Preparing Samples

Nano probe sample tubes are precision glass tubes containing a volume of approximately 40 μl of sample. A drive ring is glued onto the end, and the tube is plugged to prevent the sample from leaking. [Figure 37](#) shows a cross section of the Nano probe sample tube.

1. Prepare the liquid sample to be tested and use a pipet to transfer approximately 40 μl into the sample tube.

These sample tubes have small cavities (see [Figure 37](#)) that are easily reached with standard Pasteur pipets. A 40 μl liquid sample should just fill the bubble cavity within the sample tube. If the sample extends into the constricted region (neck) of the sample tube, the sample plug may have insufficient space to be fully inserted. A sample plug that is not fully inserted can cause spinning problems, but a small amount of plug extension may be acceptable.

Sample tubes can have a volume variability of up to 10 μl , so an alternative method of filling the sample tube is to fill the tube cavity with a known quantity of sample (e.g., 20 or 30 μl) and fill the remainder of the tube cavity with solvent.

2. Carefully insert a sample plug into the sample tube hole and firmly press it all the way in.

Be careful to not bend the sample plug, since this will make insertion more difficult. Twisting the plug during insertion can help.

The sample plugs have two regions of tight fitting diameters to achieve redundant sealing when the plug is inserted all the way. Clean up any excess sample that may have squeezed out.

Eliminating a small bubble from the sample is difficult, but careful sample metering and subsequent spinning in a centrifuge may minimize the bubble. A bubble

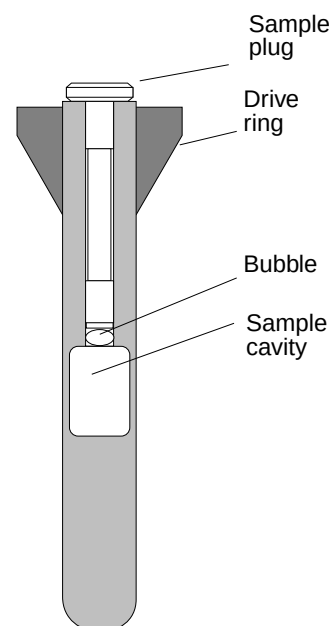


Figure 37. Nano Probe Sample Tube Assembly Cross Section

decreases the amount of sample in the tube (thus decreasing the signal intensity) but usually has little effect on the NMR otherwise.

3. Insert the sample tube into the rotor drive ring, with the flat end of the drive ring at the same end as the access hole of the sample tube.

- a. Check the location of the drive ring relative to the sample cavity by inserting the sample tube (with drive ring) into the sample depth gauge, as shown in **Figure 38**.

When the ring is positioned properly, the sample tube cavity should be aligned with the hole in the gauge.

- b. Identify the distance from the flat end of the sample tube to the flat side of the drive ring (it should be about 0.50 mm (0.020 inch)).

- c. Remove the tube from the gauge and push the ring down onto the shaft of the tube approximately 3 mm (1/8 inch). Apply a small amount of Eastman 910 (Permabond) cement onto the tube (be careful to not get cement on yourself—it can bond with skin).

The preferred method is to squeeze a small amount of the cement onto a

disposable surface, then transfer a minute quantity onto the sample tube, approximately 1 mm from the squared-off end (the end the plug is inserted into) using a toothpick.

- d. Slide the drive ring up until the flat end of the ring is the same distance from the end of the sample tube, as found previously when using the depth gauge, slightly rotating the drive ring to distribute the glue.

Note that positioning the ring incorrectly relative to the end of the sample tube results in the sample not being located totally within the observe coil. Since the sample is totally contained within the coil, incorrect positioning results in a decrease in sensitivity.

Applying a small amount of Eastman 910 to the juncture of the plug and sample tube (see **Figure 38**) might help slow down solvent evaporation.

Drive rings may be reused. To remove a drive ring that is glued onto a sample tube, place the sample tube upside down onto a flat surface and firmly press on the drive ring. The drive ring should detach from the sample tube without the need for excessive force.

4. The drive rings should already be marked with a half-black, half-white stripe. If these marks become damaged, they may be touched up with the ink and paint supplied. Make sure that the colors are applied in thin layers to avoid un-balancing the sample tube assembly.

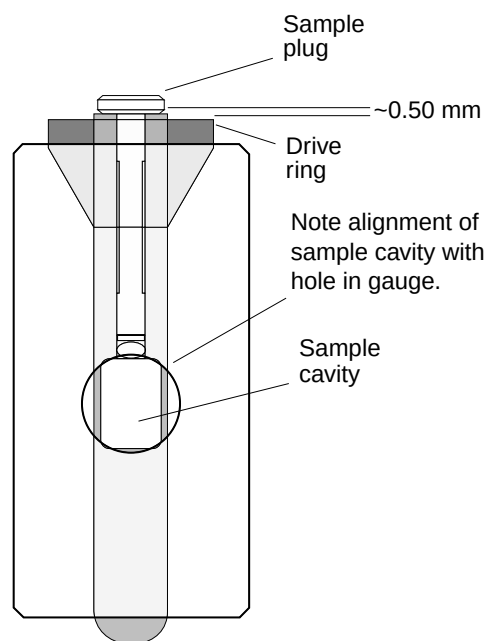


Figure 38. Nano Probe Sample Depth Gauge with Sample Tube

5. It is a good idea to spin test samples as follows with the probe outside of the magnet before attempting experiments:

- a. With the probe in the probe stand, connect the drive gas and tachometer cable as described in the section [“Connecting to UNITY-Series or VXR”](#) page 95.
- b. Ramp up the pressure and observe the spin rate.

A natural spin rate barrier exists at about 350 Hz; some samples exceed this rate at 10 psig and go on to spin well at greater than 2 kHz, while others may stay near this barrier until about 30 psig is reached.

If 30 psig is exceeded and the rotor still refuses to spin up beyond 1 kHz, drop the pressure to about 10 psig and slowly raise it again. Often, lowering the pressure and then raising it allows the sample to settle into good spinning. If this doesn't work, stop the spinning and remove the sample. Inspect the sample for any materials that may have attached to the tube. If the tube appears to be clean, detach the glue joint between the drive ring and the sample tube (see [step 3](#) above), rotate the drive ring to a new position, and try spinning again. If all else fails, try a new drive ring or a new sample tube.

4.6 Resin Bead Samples

The following steps describe a method for filling nano sample tubes with resins:

1. Insert dry resin beads into the sample tube using a Pasteur pipette as a kind of scoop.
2. Use either a necked-down Pasteur pipette (having a smaller diameter than the sample tube opening) or a 10 uL syringe to add the solvent as desired.

Remember that the resin beads can swell when you add solvent, so you might want to fill only about 15% of the sample tube with beads and the rest with solvent.

Underfilling sample tubes is better than overfilling sample tubes. Bubbles are usually of no consequence with the Nano probe.

Remember to wipe off the outside of the sample tube. Fingerprint oils and water are easily detectable with NMR if the sample is dilute and the signal average is long.

4.7 Sample Spinning

Spinning irregularities can be caused by asymmetry in the sample tube, usually related to the drive ring. Chipped or otherwise damaged sample tubes can also cause spinning problems.

You may find that some drive rings seem to spin better than others, and you may want to reuse those rings for most of your experiments. For this reason, *do not overglue the drive ring onto the sample tube*. You just want enough so that the ring will not come off while the sample is spinning. Also, if the ring is too loose on the tube, it will probably be out-of-balance when glued.

These drive ring and sample tube are designed with 4-place dimensions to help ensure trouble-free spinning, but variations within the tolerances might still occur. Typical spin rates are around 2000 Hz, but you may want to spin at higher or lower spin rates, depending on your experiment. Spin rates faster than 1000-1500 Hz help to get good line narrowing and low-intensity sidebands. You can adjust the spin rate to move spinning sidebands out of a particular region of interest or to see if a suspicious peak moves.

A vibrational hump separates the high-speed regime (about 1500 Hz) from the low-speed regime (say 400 Hz). In the low-speed regime, spinning is usually unstable; therefore, ramp up the pressure and spin fast. Do not exceed 30 psig as measured at the pneumatics box. With 30 psig (or less) the probe should be able to spin samples at 2500 Hz. Spinning a sample at 2000-2500 Hz at a pressure of 12-16 psig is ideal, since this usually indicates very smooth performance. You probably don't need to spin samples at greater than 3 KHz, but Nano samples have been spun up to greater than 4.5 KHz. Probe damage can occur if the air pressure is too high, and there is some concern that a sample tube could shatter at extremely high spin rates. This has never been observed in any Nano probe, but it is conventional wisdom for all MAS probes.

Sample spinning often has different *spinning modes*. Sometimes a sample might only spin to 400, 800, or 1800 Hz, no matter how much air you supply. If this happens, you can usually hear the probe buzz and can feel the vibration if you touch the probe body. The following steps usually fix this problem:

1. Decrease the air flow slightly, sometimes several times in a row, sometimes slowly or irregularly, and many times the sample will *catch* and take off spinning faster, quieter, and more smoothly.

The sample is spinning well if you hear a smoother, higher-pitch whine (instead of a more erratic buzzing sound) as it takes off to high speed.

If decreasing the air flow does not make the sample spin smoothly, go to the next step.

2. Turn the air flow on and off by rapidly moving the air flow knob. Sometimes the sample catches and begins to spin smoothly.

If the sample still fails to spin smoothly, go to the next step.

3. Take the sample out and rotate the drive ring relative to the sample tube about 90 degrees (a quarter turn), and try spinning the sample again.

If the sample still fails to spin smoothly, go to the next step.

4. Try a different drive ring, one that has previously proven reliable.

Sometimes the sample spins smoothly, but the tachometer does not register a spin rate, or it registers a harmonic or multiple of the spin rate. Fix this by adjusting the potentiometer on the side of the VT pneumatics/tachometer box (the one with the three air-control knobs) to get a stable, reliable reading. If you have a two-knob box (the RT pneumatics/tachometer box) you may need to pull the fiber-optic connectors of the probe out of the sockets in the box slightly, although this can be tricky and non-reproducible.

4.8 Installing Nano Sample Plugs Using the Plug Tool

Nano sample plugs may bend when inserted into the sample tubes. To avoid this problem, use the plug tool illustrated in [Figure 39](#). This tool has two usable ends, one thicker than the other. The tool acts like tweezers to grab the plug when finger clamping pressure is applied. It supports the plug while the plug is being pushed into the tube.

1. Start the plug into the hole in the sample tube.
2. Lightly grasp the plug with the tool using the thicker end.
3. Put your index finger over the head of the sample plug and push it directly into the sample tube, until the bottom of the plug head touches the tool surface.
4. After the plug has been inserted as far as possible using the thick end of the tool, remove the tool, flip it around, and grasp the plug with the thin end of the tool.
5. With your finger, push the plug straight into the sample tube until the plug head touches the tool surface.
6. Remove the tool and push the plug in the rest of the way.

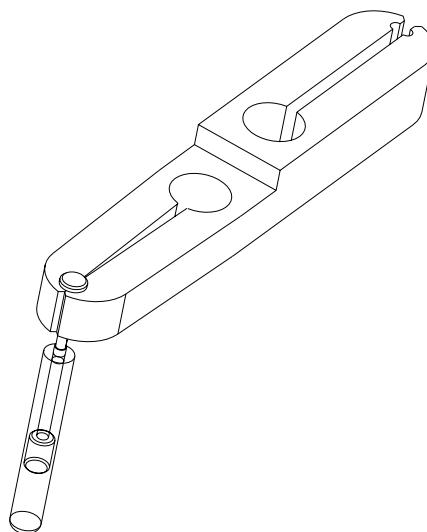


Figure 39. Plug Tool for Inserting Nano Plugs Into Sample Tubes

4.9 Probe Tuning

Tuning is adjusted with the smooth and knurled knobs extending from the bottom of the probe, as illustrated in [Figure 40](#). For best performance from the carbon Nano probe, follow the ^1H channel and X channel tuning procedures below every time you change samples.

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the red PROTON inset label turns the tuning capacitors and the smooth lower knob turns the match capacitor (see [Figure 40](#)). Turning the knurled knob clockwise (as viewed from the bottom of the probe) decreases tune capacitance, while turning the lower (smooth) knob clockwise increases the match capacitance.

Choose one of the following tuning procedures:

- *UNITYINOVA*, *UNITYplus* – See [Section 2.3](#), “Tuning Probes with *UNITYINOVA* or *UNITYplus*,” on page 33.
- *MERCURY-VX*, *MERCURY*, *GEMINI 2000* – See [Section 2.4](#), “Tuning Probes with *MERCURY-Series* or *GEMINI 2000 Systems*,” on page 37.
- *UNITY*, *VXR* – See [Section 2.5](#), “Tuning Probes with *UNITY* or *VXR*,” on page 40.
- *Gemini* – See [Section 2.6](#), “Tuning Probes with *Gemini Systems*,” on page 43.

CAUTION: Do not force the tuning capacitors to turn past the end of their ranges. The end of the range is indicated by an increase in torque and not necessarily a stop to the rotation. Forced turning can cause serious mechanical damage to the probe, requiring factory repair.

Tuning the ^1H Channel

Alternately adjust the match and tune knobs to find the minimum reflected power on the magnet leg tune meter. The knurled knob with the red PROTON inset label turns the tuning capacitors, while the smooth lower knob turns the match capacitor (see Figure 40).

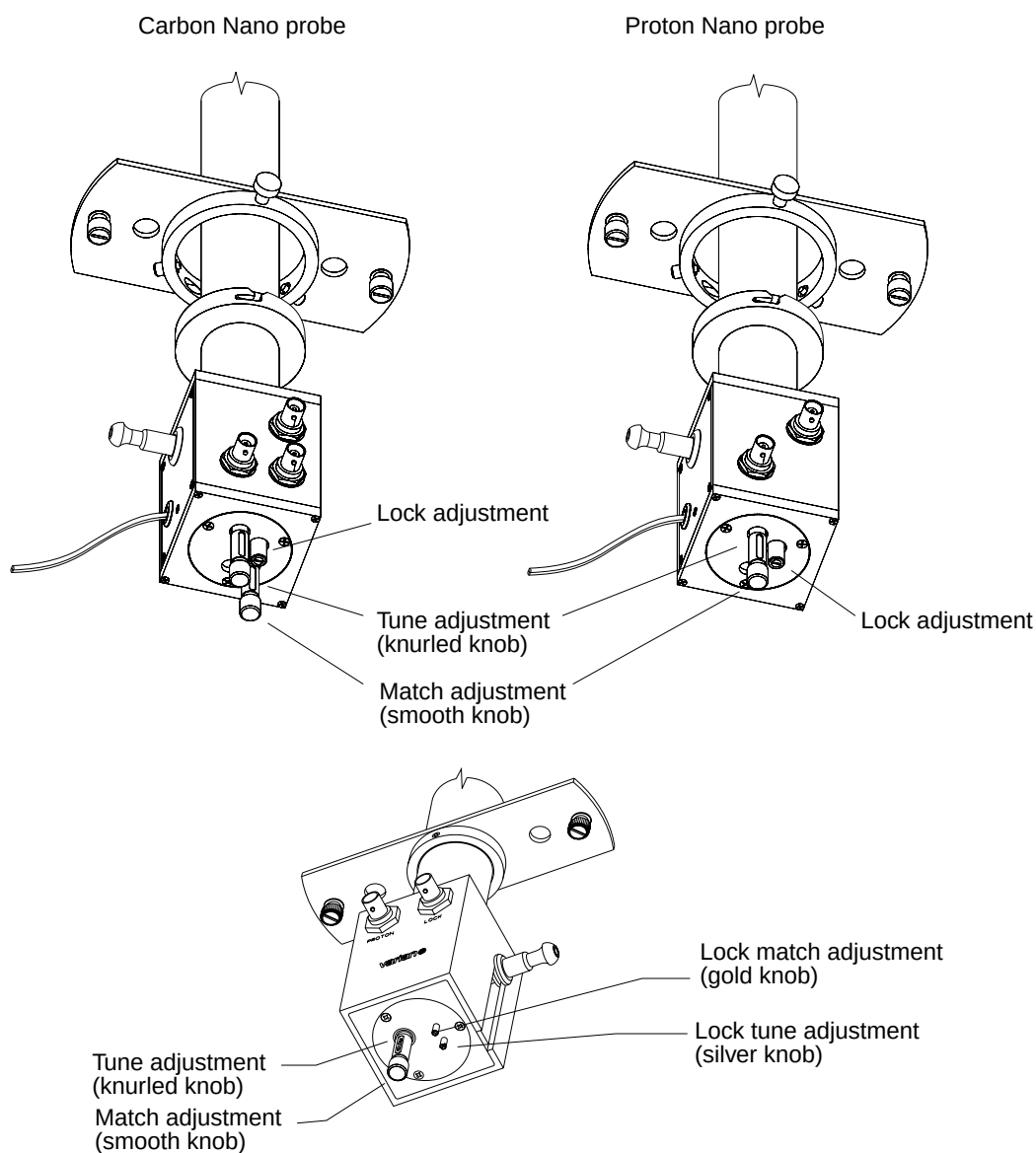


Figure 40. Nano Probe Tuning Controls

Tuning the X Channel

Turn the knurled X CHANNEL knob, shown in [Figure 40](#), to adjust the carbon tuning. To fine tune, alternately adjust the smooth and knurled X CHANNEL match and tune knobs to find the minimum reflected power on the tune meter.

4.10 Returning to Normal Operation

After tuning the probe, enter `dm='n' su` to make sure the decoupler is not inadvertently left on. Check ^1H channel tuning after X-channel tuning as well as after a sample change.

4.11 Shimming the Nano Probe

Nano probe shimming is very different from typical liquids shimming. Initially, the shimming of this probe may seem difficult, but after a little experience, shimming will seem no more difficult than shimming any other probe. Nano probes are shimmed only when the sample is spinning.

1. Insert one of the following doped samples:
 - For proton observe Nano probes, use doped D_2O .
 - For carbon observe Nano probes, use doped ASTM.
2. Install the probe as described in [Section 1.3, “Installing the Probe and Upper Barrel,” on page 18](#)).

If the Nano probe is equipped with the bayonet type of mounting flange, install the probe as described in [Section 4.1, “Using the Bayonet Probe Flange,” on page 94](#).

3. Select one of the following spin rates:
 - For proton probes, spin the sample as fast as you want without exceeding the allowed pressure setting. Between 2500 Hz and 3500 Hz is usually fine.
 - For carbon probes, spin the sample at about 1800 Hz.
4. Tune the probe and establish lock by adjusting Z0.
5. Increase the lock power and lock gain to increase lock signal without saturation.

Saturation can be detected by changing the lock power to a setting 6 dB lower than the previous value and seeing if the lock level halves. If a 6 dB lower setting does not change the lock power downward by about 50%, then lock power was set too high to begin with. In addition, too high a setting will likely result in an appearance of strobing, where the lock level fluctuates rapidly by large amounts. Proton Nano probes usually end up with lock power settings of less than 10, while carbon Nano probes require higher lock power settings.
6. Adjust the lock phase to maximize lock level.

Sometimes this adjustment is best done after some shimming has occurred, so you may want to come back to this point.
7. Start with your best shim set.

An established set for a 10-mm probe is a good starting point for the Nano probe.
8. Using only the *radial* shims (i.e., X, Y, ZX, and ZY), adjust the shims to maximize lock level.

9. Find the 90-degree pulse width value.
10. Change samples to your preferred shimming sample.
Acceptance tests for the proton Nano probe typically require the chloroform sample (Varian part no. 00-993143-99). The carbon Nano probe requires the undoped carbon sensitivity sample (Varian part no. 00-993143-69).
11. Retune the probe and set the 90 pulse width value.
12. Shim the Nano probe while considering the following:
 - In most cases, shimming is done only by adjusting four shims, namely X, Y, ZX, and ZY. Depending upon the rotational location of the probe in the magnet, one set of two shims will dominate, like X with ZX, or Y with ZY.
 - If X and ZX are the dominant shims, adjust X and compensate with ZX, always trying to increase the lock level. Adjusting ZX usually affects the base of the spectral line in a similar fashion to what you would expect with Z4 adjustments in a conventional liquids probe.
 - Avoid making axial shim adjustments, which typically make matters worse. Once you become familiar with Nano probe shimming, and understand the behavior of the probe, you can make small adjustments with Z1 and Z2, *only if necessary*.

If you are getting nowhere with your shimming, you might want to search around for another *sweet spot*. One way of doing this is to set all your shims to zero and start from scratch. If the state of shim could be viewed as a continuum, you would probably see several peaks satisfying the conditions of good shimming.

Strong spinning sidebands (e.g., 300 Hz away from the central peak) might indicate bad spinning. As mentioned in the section [Section 4.7, “Sample Spinning,” on page 101](#), dropping the pressure and then ramping the pressure up again will often cure spinning problems.

4.12 Performing an Experiment

The Nano probe must be outside the magnet before the sample can be changed. Sample tubes must be clean and have no particles of any kind adhering to them.

1. Carefully insert the bottom of the sample into the stator through the open window in the shield.
The sample should easily drop into place with the convex part of the drive ring stopping against the concave part of the stator.
2. Insert the probe into the magnet and connect the cables and pneumatics line, as described earlier for this probe.
3. Start spinning by turning up the pressure regulator until the tachometer reads the desired speed. Do not exceed 50 psig. Typically, a pressure of 30 psig should result in a spin rate of greater than 2 kHz. If the Pneumatics/Tachometer Box does not register any speed, but the sample is obviously spinning, check the paint markings on the drive ring and make sure the fiber optic connectors are properly connected (only handle the fiber optic cable by the black connectors). If you have a VT Pneumatics/Tachometer Box, you can try adjusting the potentiometer until the proper speed is registered.
4. Tune the probe, while the sample is spinning, as described earlier in this section.

5. Shim the magnet as described earlier in this section and take acquisitions as necessary.

4.13 Changing Samples or Solvents

1. Decrease the pressure gradually until sample rotation ceases.
If the sample had been spinning at 2 kHz, allow about 30 seconds to one minute to ramp the speed down.
2. Detach the pneumatics line at the probe, and remove the probe from the magnet. The tachometer lines may be left attached to the Pneumatics/Tachometer Box if the probe is not to be moved far from the magnet.
3. Tilt the probe and lightly tap on the shield until the sample falls out into your hand, or carefully pull the sample out with your fingers.
4. Put the new sample in and continue as before. Keep the samples stored in the appropriately marked vials in the probe and rack stand.

4.14 Choosing a Sample Cell Filling Device

Several devices can be used to fill the Nano probe sample cell:

- Standard glass Pasteur pipette fits into the Nano probe sample tube, but the pipette has no graduation, which makes difficult transferring precise amounts difficult.
- Costar[®] plastic pipette tips (10800-492-1110, catalog No. 4853, rounds, elongated, 1 to 200 μ L, tip diameter 0.5 mm) fits into a Nano probe sample tube and is repellent (nonwetting) with enough solvents to typically transfer the entire sample.
- A standard Hamilton syringe works well—with a metal needle and plunger (10 μ L syringe, J&W Scientific part no. 705-2001); with a metal needle and gas-tight plunger (10 μ L syringe, J&W Scientific part no. 705-2112).
- Large-capacity Teflon needles work well—90 μ L capacity Teflon needle (Wilma part no. 90622; Sigma part no. Z11735-8, 12-inch, 14-gauge).

Large-capacity Teflon needles must be used with Luer-Lock syringe, preferably a gas-tight model such as 500- μ L Hamilton Luer-Lock Gas-Tight syringes (Wilma part no. 81220).

These long needles, especially when equipped with larger-volume syringes, are good for washing out or *irrigating* Nano probe sample cells. Resin slurries, even if made in nonpolar solvents, can often be fully removed with a jet of tap water delivered with this long-needle, high-volume syringe combination.

4.15 Using the Sample Cleaning Holder

The Nano sample cleaning holder allows Nano sample tubes to be held upside-down to facilitate cleaning. In use, tubes are held by friction in the holder, which is clamped so that the tubes can be syringed with solvent, the solvent dripping into a beaker held underneath.

1. Clamp the 1/2-inch rod in a standard laboratory clamp so that the holes in the black plastic part point downward.

2. Push the sample tubes to be cleaned into the holes in the black plastic part so that the tube opening faces downward.
The tubes do not need to be fully inserted--just enough to hold them upside-down.
3. Syringe cleaning solvent into the tubes until clean.
4. Allow tubes to dry and remove.

Chapter 5. Probes for Solid-State NMR

Sections in this chapter:

- 5.1 “Rotor Composition” [this page](#)
- 5.2 “Sample Spinning” [this page](#)
- 5.3 “Adjusting the Sample Angle to the Magic Angle” [page 113](#)
- 5.4 “MagicAngle Probes for VT and RT CP/MAS” [page 117](#)
- 5.5 “MagicAngle Probes for RT CP/MAS” [page 123](#)
- 5.6 “Wideline Probes” [page 126](#)
- 5.7 “Multipulse Probes for CRAMPS” [page 129](#)
- 5.8 “Doty VT CP/MAS Probes” [page 131](#)

This chapter covers the many types of solid-state NMR probe. Connecting the cables, tuning the probe, tuning ranges, sample spinning, and any special information specific to the probe is covered in each section.

5.1 Rotor Composition

Rotors for solid-state NMR are made of partially stabilized zirconia (PSZ) or silicon nitride with pMMA or Torlon caps, vented and nonvented versions. The cap twist-fits into the rotor body and is held with two O-rings. For more information on preparing CP/MAS samples, refer to the chapter on solid-state NMR in the manual *System Operation*.

Current Si_3N_4 rotors are of two-piece construction. If the rotor plug should break loose from the rotor body, the plug may be reinserted with cyanoacrylate if quickly pressed into position.

5.2 Sample Spinning

On high-speed rotors, tachometer sensing is on the rotor bottom. Zirconia rotors are marked with a permanent black marking pen or black enamel paint so that 50% of the bottom of surface area is shaded black; silicon nitride (Si_3N_4) rotors are marked with white enamel paint in the same fashion. Centrifugal force and rotor crashes can cause the black and white markings to flake off around the edges. This can cause inaccurate tachometer readings. The black or white half circle can be reapplied on the rotors. The diameter marking should be straight.

Doty high-speed rotors have optical markings inside the bottom cap; other Doty probes use electrostatic sensing (triboelectric). See the Doty manual for instructions on reapplying the optical marking.

Varian high-speed rotors (with and without vent holes) are composed of zirconia or of silicon nitride (Si_3N_4) with pMMA or Torlon (type 4203) end caps. Always check the maximum spin rate specification for Varian rotors and end caps as listed [Table 19](#).

WARNING: A projectile hazard exists if a spinning rotor explodes. To prevent possible eye injury from an exploding rotor, avoid spinning rotors outside the magnet. If it is necessary to spin a rotor outside the magnet, use a certified safety shield and full face shield at all times. Never use rotors that have been dropped onto a hard surface, since microcracks in the rotor material can cause rotor explosions at much lower spinning speeds than indicated in [Table 20](#) for 7-mm probes and in [Table 21](#) for 5-mm probes. Never spin 7-mm zirconia (white) rotors at spinning speeds above 7.2 kHz. Never spin 7-mm silicon nitride (gray) rotors at speeds above 9.5 kHz. Never spin 5-mm zirconia (white) rotors with a Varian probe. Never spin 5-mm silicon nitride (gray) rotors at speeds above 15 kHz. Never apply air drive pressure above 72.5 psig (5.0 bar).

Table 19. Characteristics of Varian Rotors and End Caps

Item	Material	Color	Recommended Temperature Range (C)	Max Spin Rate Ambient (Hz)	Max Spin Rate VT (Hz)	Varian Part No.
Rotor, 7 mm	Zirconia	white or off-white	—	7200	—	00-991036-00
Rotor, 7 mm	Silicon nitride	gray	−125° to +125°	9500	9000	00-990874-00
Rotor, 5 mm	Silicon nitride	gray	−125° to +125°	15000	13000	00-992521-00
Rotor, 5 mm	Silicon nitride	gray (CRAMPS)	−125° to +125°	15000	13000	00-993263-00
Cap, 7 mm	Torlon	green	−125° to +125°	9500	9000	00-991646-01
Cap, 7 mm	Torlon, vented	green	−125° to +125°	5000	—	00-991647-00
Cap, 5 mm	Torlon	green	−125° to +125°	15000	9000	00-992519-00
Cap, 5 mm	Torlon, vented	green	—	5000	—	00-992524-00
Cap, 7 mm	pMMA	colorless-clear	−125° to ambient	9500	9000	00-991038-00
Cap, 5 mm	Kel-F, vented	colorless-opaque	−125° to + 80°	5000	5000	01-902171-00
Cap, 7 mm	Kel-F	colorless-opaque	−125° to + 80°	7000	7000	00-992558-00
Cap, 5 mm	Kel-F	colorless-opaque	−125° to + 80°	7000	7000	00-993228-00
Cap, 5 mm	Kel-F	colorless-opaque	−125° to + 80°	7000	7000	00-993262-00

Spinning Samples in Doty CP/MAS Probes

Either one or two air supplies can be used for sample spinning in the Doty probe. Because the control box supplied for CP/MAS has two controllers, split streams are recommended. The techniques for doing this are covered in the Doty probe manual.

Spinning Samples in High-Speed CP/MAS Probes

Spin rates and the bearing and drive pressures for the Varian VT CP/MAS probe at ambient temperature are listed in [Table 20](#) for the 7-mm probe and [Table 21](#) for the 5-mm probe.

Note that for 5-mm CP/MAS probes, spinning a sample with a small amount of drive gas before applying the initial 28 psig (2.0 bar) of bearing pressure is sometimes useful. The rotor will begin spinning and will therefore become less likely to flutter (slight motion in and out of the stator) during initial spin up.

To avoid rotor explosions, never spin 7-mm zirconia rotors faster than 7200 Hz and never spin 5-mm zirconia rotors in Varian probes. Also, never spin 7-mm silicon nitride rotors

Table 20. Typical 7-mm VT CP/MAS Probe Spin Rates With Bearing and Drive Values

<i>Spinning Speed*</i> (Hz $\pm$ 250 Hz)	<i>Bearing</i>		<i>Drive</i>	
	<i>Pressure</i> psig (bar)	<i>Flowrate</i> (LPM $\pm$ 2 LPM)	<i>Pressure</i> psig (bar)	<i>Flowrate</i> (LPM $\pm$ 2 LPM)
2500	28 (2.0)	12.5	7 (0.5)	15.0
4000	28 (2.0)	12.5	14 (1.0)	20.0
5000	36 (2.5)	12.5	21 (1.5)	25.0
6000	36 (2.5)	12.5	28 (2.0)	27.5
7200	36 (2.5)	12.5	36 (2.5)	30.0
8000	44 (3.0)	10.0	51 (3.5)	35.0
8500	44 (3.0)	10.0	58 (4.0)	37.5
9000	44 (3.0)	9.0	65 (4.5)	40.0

* Rates are approximate values for the Varian 7-mm VT CP/MAS probe at ambient temperature. The actual spin rate will vary depending on the properties of the sample and sample holder.

Table 21. Typical 5-mm VT CP/MAS Probe Spin Rates With Bearing and Drive Values

<i>Spinning Speed *</i> (Hz $\pm$ 250 Hz)	<i>Bearing</i>		<i>Drive</i>	
	<i>Pressure</i> psig (bar)	<i>Flowrate</i> (LPM $\pm$ 2 LPM)	<i>Pressure</i> psig (bar)	<i>Flowrate</i> (LPM $\pm$ 2 LPM)
4500	28 (2.0)	12.5	7 (0.5)	15.0
7000	28 (2.0)	12.5	14 (1.0)	20.0
11500	49 (3.5)	12.5	35 (2.5)	25.0
13000	56 (4.0)	9.0	63 (4.5)	40.0

* Rates are approximate values for the Varian 5-mm VT CP/MAS probe at ambient temperature. The actual spin rate will vary depending on the properties of the sample, sample holder, and temperature.

faster than 9500 Hz or 5-mm silicon nitride rotors faster than 15000 Hz. For samples that have densities above 3.0 g/cc, decrease the maximum spin rate by 35%.

WARNING: Excessive spinning speeds can cause the rotors to shatter and explode. Never spin zirconia (PSZ) rotors (white or off-white in color) above 7.2 kHz or silicon nitride rotors (gray) above 9.5 kHz. For samples that have densities above 3.0 g/cc, decrease the maximum spin rate by 35%.

To spin samples in high-speed CP/MAS probes:

1. Using your fingers, insert either a pMMA or Torlon end-cap into the rotor to be spun. Rotate the end-cap while pushing it into the rotor. Make sure the end-cap is fully seated into the rotor.
2. Make sure the bearing and drive air pressure are off.
3. Carefully place the rotor with the end-cap into the stator and install the probe into the magnet.
4. Turn the air bearing pressure to 28 psig (2.0 bar); the rotor should start spinning slowly at 500–900 Hz.
If using a 5-mm CP/MAS probe, to avoid rotor flutter (slight motion in and out of the stator) during initial spin up, you can begin spinning a sample with a small amount of drive gas before applying the initial 28 psig (2.0 bar) of bearing pressure.
5. Slowly turn on the air drive pressure to 3.6 psig (0.25 bar) and wait for 15 seconds to allow the rotor to stabilize.
6. Gradually increase the air drive pressure to 7 psig (0.5 bar) and again wait 15 seconds. The spinning speed should gradually increase to about:
 - 2500 Hz for 7-mm probes
 - 4500 Hz for 5-mm probes
7. Slowly increase the air drive pressure to 14 psig (1.0 bar). The spinning speed should reach about:
 - 3700 Hz for 7-mm probes
 - 7000 Hz for 5-mm probes
8. If rotor speeds faster than 3700 Hz for 7-mm probes or 7000 Hz for 5-mm probes are required, slowly increase the air bearing pressure as indicated in [Table 20](#) and [Table 21](#). Then adjust the drive pressure:

It may be necessary to increase the bearing pressure for ill-behaved samples or for very high spinning speeds. Provided that the two flowmeter valves are fully open, flowmeters require no adjustment at any time. Never adjust the spin rate with the flowmeter.

WARNING: When operating under high pressure, make sure the valves on older flow meters are not too loose. Older flow meters have removable flow knobs that can fly off if loosened too much when under high pressure.

Rotor “touch down” can sometimes occur during spin-up if the rotor is unbalanced or if it has insufficient bearing gas pressure. The spinner system is designed to handle occasional touch downs. To recover from rotor touch down, quickly decrease the drive pressure, increase the bearing pressure, and slowly increase the drive pressure so that a higher spin rate can be achieved. If the problem persists, reduce the drive and bearing pressures to zero and remove and inspect the sample.

CAUTION: To prevent damage to the rotor or bearing, always smoothly shut off the rotation gas using the rotation/drive pressure regulator before using the bearing pressure regulator to turn off the bearing gas.

To remove a sample, take care to decrease the rotor speed smoothly. At all times that rotation air is flowing, bearing air should read at least 28 psig (1.9 bar). Only when the rotation/drive air is completely off should the bearing be carefully decreased to zero.

Overcoming Imbalance

Most of the spinning problems encountered with filled rotors result from imbalance caused by the sample material. A damaged rotor might be at fault but that can be eliminated by always checking the spinning quality of the empty rotor before packing it with the sample material. Discard damaged rotors.

If a packed rotor does not spin properly at first, inspect it to see if the sample has been disturbed. Part of the sample may have broken loose and been thrown out of the rotor, in which case repacking might be the solution. Sometimes, loose material balances itself if kept in the rotor and spun below its vibration speed for a few minutes. If the sample seems intact on the surface, it is probably not homogeneous or unevenly packed. In the case of a machined plug, the material can have a void or it can fit too loosely in the rotor cavity. The only solution is to remove all of the sample and repack the rotor. With inhomogeneous materials, this repacking may have to be tried more than once.

CAUTION: When removing caps or digging out packed samples, take care not to gouge the rotor. Even small scratches can imbalance the rotor.

At times, worn rotor caps cause imbalance. Changing caps or rotating them between rotors sometimes cures these problems.

Increased bearing pressure often stabilizes samples that do not spin well. This adjustment must be made at low speed and then ramped up once the rotor is spinning stably.

Rotor Synchronization

If the spectrometer is equipped with a rotor synchronization option (Part No. 00-990385-00), the spinning speed can be read by the acquisition system.

Entering `hsrotor='y'` `su` shows the current spinning speed in the acquisition status window. The parameter `srate` is updated after each experiment to show the spinning speed. Furthermore, if `in='y'`, an acquisition is halted if the spinning speed differs by more than 100 Hz from spinning speed at the start of the acquisition.

For further details on rotor synchronization, refer to the manual *System Operation*.

5.3 Adjusting the Sample Angle to the Magic Angle

The magic angle is set at the factory. Little or no adjustment is required in the field.

Coarse Adjustment

A convenient method of setting the sample angle to the approximate magic angle before final optimization with NMR is to use the angle measuring stem (Part No. 00-992825-00)

together with the angle measuring gauge (Part No. 00-992826-00) from the rotor and tool kit. **Figure 41** illustrates how the angle measuring stem and angle gauge are used.

Fine Adjustment

The preferred method of adjusting the magic angle uses the ^{79}Br spectrum of KBr, which has a large chemical shift anisotropy (CSA). When spun at the magic angle, this results in an extensive set of spinning sidebands. As ^{79}Br is very close in frequency to ^{13}C , it is easy to switch between the two nuclei.

The magic angle can easily be precisely set (or checked) as follows:

1. Enter `tn= 'Br79'` to obtain the parameter set to observe ^{79}Br .

If the message appears that Br79 is not an entry in the nucleus table `nuctabXX` (where `XX` is 2d, 3d, 4d, or 5d), add the ^{79}Br nucleus using the `vi` text editor (familiarity with this UNIX text editor is assumed). From VNMR enter:

```
vi( '/vnmr/nuctables/nuctabXX' )
```

where `XX` is 2d, 3d, etc. (e.g., `vi( '/vnmr/nuctables/nuctab2d' )`)

Then, insert one of the following lines above the Br81 line:

- 200-MHz systems – Insert:

```
Br79 50.180 1.480e6 low yes 0.0
```

- 300-MHz systems – Insert:

```
Br79 75.180 1.480e6 low yes 0.0
```

- 400-MHz systems – Insert:

```
Br79 100.208 1.4842e6 low yes 0.0
```

Exit the `vi` editor and then enter `tn= 'Br79'` again. No error should be generated.

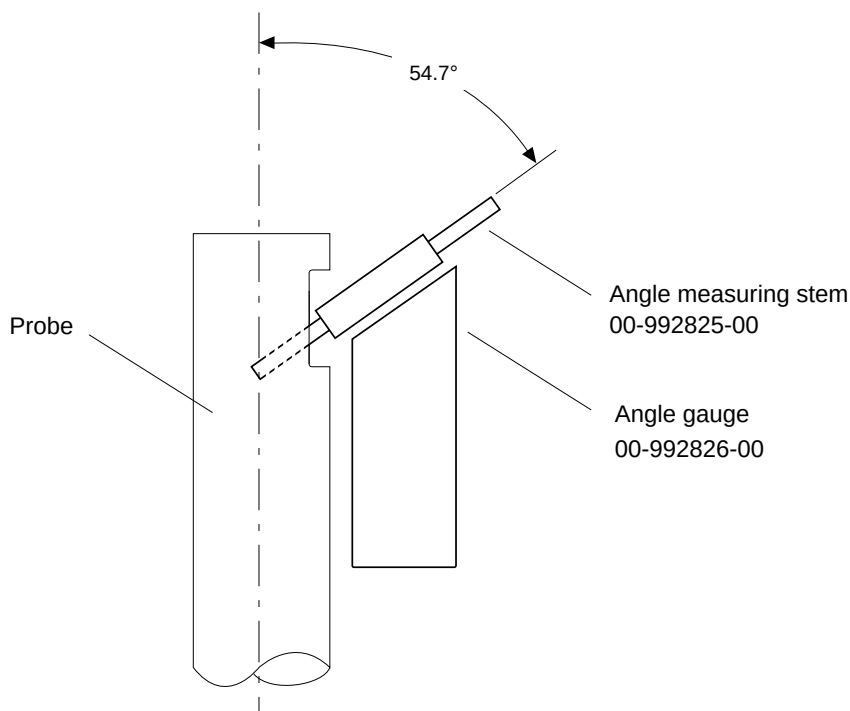


Figure 41. Tools for Coarse Adjustment of Sample Angle

2. Load a rotor with KBr, insert it in the probe, spin at 3 kHz, and tune the system for ^{79}Br . Grinding the KBr crystals before packing the rotor is helpful.
3. Set `seqfil='s2pul'` `sw=1e5` `at=0.02` and obtain a single transient spectrum by entering `ga`. Set the cursor on resonance, and enter `movetof`.
4. Set `phfid=0` and enter `gf`.
5. Open the `acqi` window, click the FID button, and observe the real-time FID display.
The FID displays a transient consisting of an exponential decay with a “picket fence” of one or more spikes (see Figure 42 and Figure 43). If the signal is not exactly on resonance, adjust `Z0` until it is, then select IPA and adjust `phfid` to maximize the on resonance FID.
6. Adjust the angle using the appropriate method below:
 - *Varian RT CP/MAS probes* – Turn the screw between the two copper coax lines in the probe baseplate.
 - *Varian VT CP/MAS probe* – Turn the fiberglass rod with the adjustment tool.
 - *Doty CP/MAS probe* – Turn the smaller gold rod.
 Maximize the size and number of spikes in the picket fence. The spikes should persist for about 10 ms. The sample angle is now set to the magic angle.
7. Close the `acqi` window and retune the probe to the appropriate nucleus.

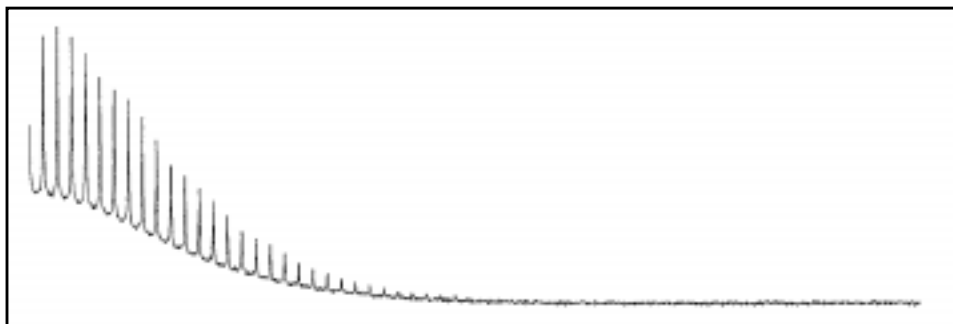


Figure 42. FID Display of KBr On-Angle

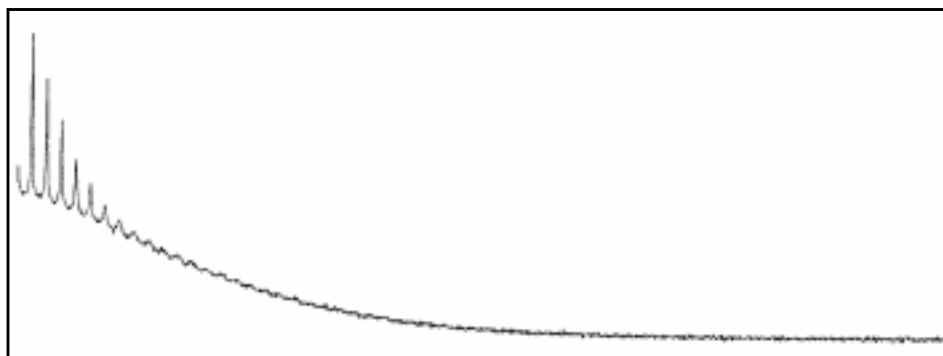


Figure 43. FID Display of KBr 1/2 Turn Off-Angle

The same sample and general procedure can be used to monitor spinning stability, both in angle and in speed. An angular instability shows in two ways: the shape and size of the exponential varies from transient to transient, and the picket fence shows instabilities in length and amplitude if the rotor is vibrating.

Lack of stability in the spinning speed can be measured by inspecting the summed FID. If acquisition is continued for a time, the speed variation can be determined from the broadening of pickets well down the fence in time. Run a four-transient FID and turn the FID into a “spectrum” by entering `ft('nof t')`. The FID now displays as a spectrum, so you can use cursors and related commands to edit the display. Measure the resolution of a picket at the start and the end of the FID display. Similar values indicate good spinning stability.

An alternative method of adjusting the magic angle is to use ^{13}C CP/MAS of hexamethylbenzene (HMB), the standard sample, which has two ^{13}C resonances. Of these, the aromatic carbon line (on the left side of the spectrum) is extremely sensitive to the angular adjustment. **Figure 44** shows a typical spectrum, which includes the sidebands, of the aromatic resonance. Adjust the aromatic line for minimum linewidth and maximum intensity.

In general, the angle should stay fairly constant; however, this is not guaranteed. The angle should be checked and adjusted:

- When the probe is inserted in the magnet.
- Every day or second day if continuing operation.
- If linewidths in any particular sample are suspiciously large.

Once typical values for the minimum linewidths are established for any particular instrument, these values can be taken as a reliable indication of proper angle. Adjustment of the angle is neither necessary nor desirable if the first measurement indicates that the minimum linewidth has been achieved.

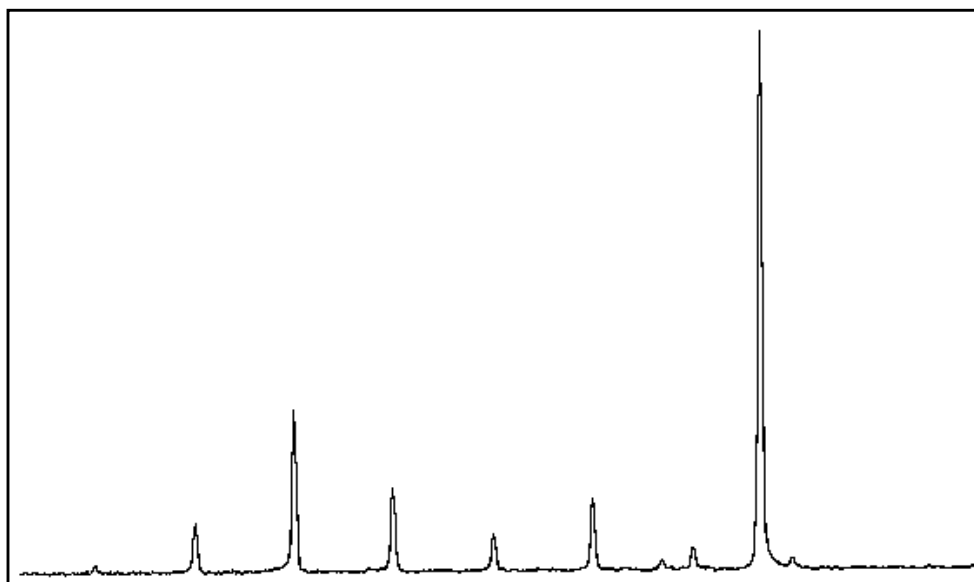


Figure 44. Typical Hexamethylbenzene (HMB) Spectrum

5.4 MagicAngle Probes for VT and RT CP/MAS

This section covers the Varian VT (variable temperature) and RT (room temperature) CP/MAS probes listed in the following table. The RT versions use the same body style and function the same as the VT version, except without the VT capability.

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>VT or RT</i>	<i>Sample Size (mm)</i>	<i>Tune Range (MHz)</i>	<i>Probe Part No.</i>
300	51	VT	7	45–123 (^2H – ^{31}P)	00-969336-00
300	51	RT	7	45–123 (^2H – ^{31}P)	00-969336-00
300	89	VT	5	45–123 (^2H – ^{31}P)	00-992318-00
300	89	VT	7	45–123 (^2H – ^{31}P)	00-969336-01
300	89	RT	7	45–123 (^2H – ^{31}P)	00-969336-01
300	89	VT	5	45–123 (^2H – ^{31}P)	00-992318-01
400	54	VT	7	60–163 (^2H – ^{31}P)	00-969337-01
400	54	RT	7	60–163 (^2H – ^{31}P)	00-969337-01
400	54	VT	5	60–163 (^2H – ^{31}P)	00-993219-01
400	89	VT	7	60–163 (^2H – ^{31}P)	00-969337-01
400	89	RT	7	60–163 (^2H – ^{31}P)	00-969337-01
400 ¹	89	VT	7	40–162 (^{15}N – ^{31}P)	00-992360-00
400	89	VT	5	60–163 (^2H – ^{31}P)	00-992319-01

1. Widebody probe.

The section contains the following subsections:

- “Connecting the Probe,” [this page](#)
- “Installing ^{15}N – ^{31}P MagicAngle Probes” [page 119](#)
- “Tuning Ranges for VT and RT MagicAngle Probes” [page 120](#)

Connecting the Probe

The following steps outline the procedure for connecting the probe, filters, and pneumatics/tachometer box (see [Figure 6](#)). Also in these instructions are steps for tuning the decoupler and observe channels when using the probe with a UNITY or VXR spectrometer. To tune the probe when using it with a UNITY INOVA or UNITYplus system, refer to [Section 2.3](#), “Tuning Probes with UNITY INOVA or UNITYplus,” on page 33. These steps should be completed before running a CP/MAS experiment.

1. Slide the upper barrel up about 50 mm (2 inches).
2. Place the rotor in the probe. Insert the probe into the magnet.

Varian VT CP/MAS probes have an adjustable bottom flange. The position of the flange is shown in [Figure 45](#). The collar near the top of widebore versions is for centering the probe in the magnet. The distance H, in [Figure 45](#), is from the top of the flange to the two screws in the probe shield. [Table 22](#) shows the distances for the different probes and magnets. Capacitor and inductor sticks are provided to extend the tuning range.

To install the ^{15}N – ^{31}P VT CP/MAS probe, refer to the section “Installing ^{15}N – ^{31}P MagicAngle Probes” [page 119](#).

3. Connect the BEARING air supply from the pneumatics and tachometer box to the BEARING port of the probe.
4. Connect the ROTATION/DRIVE air hose to the ROTATION/DRIVE port on the probe.
5. Attach the appropriate low-pass notch filters, listed below, between the OBS connector on the probe and the PROBE connector on the preamplifier.

<i>Magnet (MHz)</i>	<i>Number of Filters</i>	<i>Filter Part No.</i>
200	2	00-948068-00
300	1	00-953049-00
400	1 or 2	00-953708-00

6. Attach the appropriate high-pass filter, listed below, to the DEC connector *on the probe*. Connect the decoupler cable from the magnet leg to the filter. Connecting the high-pass filter to the probe, instead of the preamplifier, prevents rf interference.

<i>Magnet (MHz)</i>	<i>Filter Part No.</i>
200	00-948800-00
300	00-953374-00
400	00-953768-00

7. Connect the probe fiber optic cables to the optical port on the pneumatics/tachometer box (shown in [Figure 6](#)).
8. Start the sample spinning (For more information on sample spinning, see [Section 5.2, "Sample Spinning,"](#) on page [109](#)).
9. Tune the decoupler channel on the probe as follows:
 - a. Enter `tn= 'H1' su` to set the proton frequency.
 - b. Switch the magnet leg TUNE switch to OBSERVE and connect the probe decoupler channel to the TUNE connector on the leg.
 - c. Adjust the red (tune) and yellow (match) controls for minimum meter deflection.

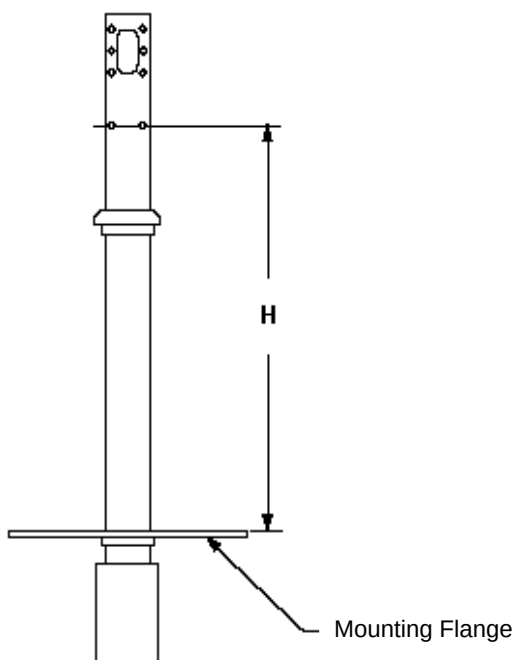


Figure 45. CP/MAS Probe Bottom Flange Position

10. Tune the observe channel on the probe as follows:
 - a. Change to another experiment and enter `tn='C13' su`.
 - b. Connect the decoupler cabling back to its normal observe configuration and connect the probe observe channel to the TUNE connector on the preamplifier.
 - c. Adjust the green (tune) and blue (match) controls for minimum reflected power.
11. Repeat steps 9 and 10 (in order) until no further improvement in tuning is achievable. When finished, set the TUNE switch back to DEC and return all cables from the probe to their normal connectors.

Table 22. Flange Positions for Varian CP/MAS Probes

<i>Magnet (MHz/mm)</i>	<i>Distance H cm (in.)</i>	<i>Magnet Part Number</i>
400/54	29.85 (11.75)	00-958230-00
400/89	38.28 (15.07)	00-953230-13
300/51	19.58 (7.71)	00-958220-01
300/89	32.72 (31.72)	00-958230-12

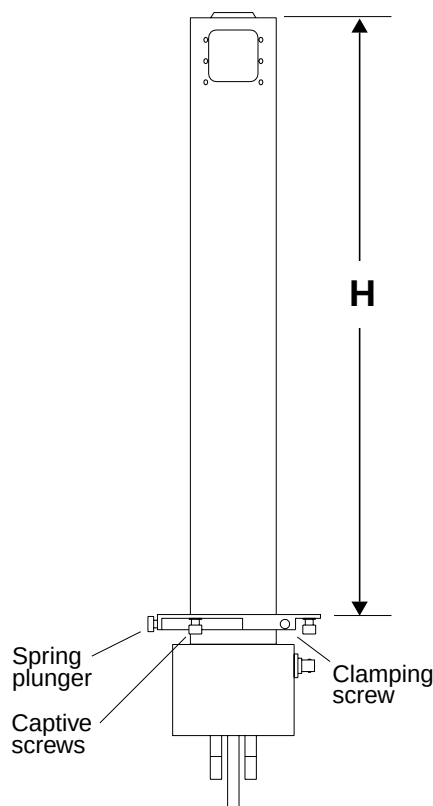
Installing ^{15}N – ^{31}P MagicAngle Probes

The Varian ^{15}N – ^{31}P VT CP/MAS probe (wide body probe) includes a probe flange designed to facilitate positioning and installation of the probe within the magnet. In addition, the flange allows the sample to be positioned at the same field location repeatedly, regardless of how many times the probe has been removed from the magnet. This probe flange is composed of two parts, a flange plate that remains attached to the magnet and the inner flange ring that remains attached to the probe.

The steps below describe how to install and remove the ^{15}N – ^{31}P CP/MAS probe and flange.

Positioning the Flange and Probe

1. Position the flange on the probe body so the distance from the top end of the probe to the top surface of the flange is 49.99 cm (19.68 in.), shown as H in [Figure 46](#).
If moving the flange is difficult, insert a Phillips screwdriver clamping screw through the hole on the perimeter of the flange plate and loosen the clamping screw.

**Figure 46.** Varian ^{15}N – ^{31}P CP/MAS Probe Flange Position

2. Once positioned, tighten the flange by inserting a Phillips screwdriver through the hole on the perimeter of the flange plate and tighten the clamping screw.

This causes the inner flange ring to clamp onto the probe body. Use only enough clamping pressure to hold the flange onto the probe body.

CAUTION: Extreme force could cause damage to the probe shield. Also, be certain that the spring plunger has “clicked” into place in the inner flange ring. Improper mating of the flange and the flange ring may cause the probe to slip out of the magnet.

3. Insert the probe into the magnet, applying a firm pressure upward until the flange makes contact with the magnet.
Note that eddy currents that build up in the large probe shield make it difficult to insert this probe.
4. Rotate the probe and flange until the captive screws on the flange line up with the holes in the RT shim set. Positioning the probe with the spring plunger on the flange to the left is preferable. Tighten the captive screws.

Removing the Probe

1. To avoid damaging the glass dewars, detach the nitrogen gas connections.

CAUTION: Keep the probe from falling out of the magnet. The probe is heavy and can drop out of the magnet if not supported during removal.

2. Pull outward on the spring plunger and rotate the probe body in a counter-clockwise direction, as viewed looking upward at the bottom of the probe.
The probe will rotate approximately 10 degrees then stops.
3. Withdraw the probe downward and out of the magnet. The flange plate remains attached to the magnet while the inner flange ring remains attached to the probe.

Installing the Probe After the Flange is Positioned

1. Slide the probe up into the magnet.
2. Insert the inner flange ring (attached to the probe) into the flange plate (attached to the magnet). Note that the setscrew nubs that protrude into the inner diameter of the flange plate must mate with the grooves cut into the outside diameter of the flange ring.
3. When the inner flange ring is fully inserted into the plate, rotate the probe slightly in the clockwise direction, as viewed upward toward the bottom of the probe.
The spring plunger should snap into place when the probe is rotated to the correct location. Note that since there is tri-fold symmetry to the flange, there are three angles of rotation where the probe may snap into place.

Tuning Ranges for VT and RT MagicAngle Probes

The nominal tuning ranges ($\pm 2\%$) for CP/MAS probes are listed in tables as follows:

- 300-MHz, 5-mm probes, see [Table 23](#); 7-mm probes, see [Table 24](#)
- 400-MHz, 5-mm probes, see [Table 25](#); 7-mm probes, see [Table 26](#)

Table 23. Tuning Range of Varian 300-MHz, 5-mm CP/MAS Probe

<i>Capacitor Stick (pF)</i>	<i>Range (MHz, $\pm 2\%$)</i>	<i>Part Number</i>	<i>Inductor Stick (Turns)</i>	<i>Range (MHz, $\pm 2\%$)</i>	<i>Part Number</i>
6	67.6 – 74.5	00-992465-02	50	79.4 – 89.0	00-992464-04
10.3	64.0 – 69.5	00-992465-03	23	84.0 – 94.3	00-992464-02
15.2	60.8 – 65.3	00-992465-00	7.5 ¹	121.4 – 121.4	
20 ²	57.3 – 61.2	00-992465-07			
23	56.0 – 59.5	00-992465-08			
38	49.5 – 51.7	00-992465-09			
51 ³	46.0 – 46.0	00-992465-xx			
No Stick ⁴	75.0 – 79.4				

1. ³¹P requires the 7.5 turn inductor stick.

2. ²⁹Si requires a 20 pF stick.

3. ²H requires the 51 pF stick.

4. ⁷⁹Br, ¹³C, and ²⁷Al can be reached with no stick.

Table 24. Tuning Range of Varian 300-MHz, 7-mm CP/MAS Probe

<i>Capacitor Stick (pF)</i>	<i>Range (MHz, $\pm 2\%$)</i>	<i>Inductor Stick (Turns)</i>	<i>Range (MHz, $\pm 2\%$)</i>
2.0	71–75	9	117–121.4
4.0	68–72	11	115–118
6.0	66– 69	12	106–117
9.2	63– 66	23	94–99
10.3	62– 65	30	91–95
13.6	60– 62	50	82–88
15.2	58–60	74	79–84
20.0	56.5–57.5	No Stick	75–78.2
23.0	52.5–53.5		
25.0	51.4		
30.0	48.2		
38.0	46.0		

- 400-MHz, ¹⁵N–³¹P (wide body) probes, see [Table 27](#)

Table 25. Tuning Range of Varian 400-MHz, 5-mm CP/MAS Probe

<i>Capacitor Stick (pF)</i>	<i>Range (MHz, $\pm 2\%$)</i>	<i>Inductor Stick (Turns)</i>	<i>Range (MHz, $\pm 2\%$)</i>
2.0	94 – 98	6	161.9
4.0	90 – 93	8	153 – 158
6.0	86 – 89	9	146 – 151
8.2	83 – 85	11	139 – 144
9.2	80 – 83	23	121 – 126
10.3	79 – 81	36	114 – 119
13.6	76.3	50	109 – 113
15.2	70.4 – 71.3	74	105 – 109
20.0	65.8	No Stick	99.2 – 104.5
25.0	64.2		
28.8	61.4		

Table 26. Tuning Range of Varian 400-MHz, 7-mm CP/MAS Probe

<i>Capacitor Stick (pF)</i>	<i>Range (MHz, $\pm 2\%$)</i>	<i>Inductor Stick (Turns)</i>	<i>Range (MHz, $\pm 2\%$)</i>
2.0	92 – 97	9	154 to 163
4.0	87 – 91	12	143 – 154
6.0	83 – 87	23	124 – 133
9.2	77 – 80	30	115 – 123
10.3	75 – 78	50	108 – 116
13.6	71 – 73	74	104 – 110
15.2	68.5 – 70.8	No Stick	98 – 105
20.0	63.6 – 65.6		
23.0	60.2 – 61.8		

Table 27. Tuning Range for 400-MHz, 7-mm ^{15}N – ^{31}P CP/MAS Probe

<i>Stick</i>	<i>Range (MHz, $\pm 2\%$)</i>
No stick	51–156
43-pF capacitor	39–50
45-turn inductor	50–165

5.5 MagicAngle Probes for RT CP/MAS

This section covers the mechanical operation of the following Varian RT (room temperature) CP/MAS probes:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Tune Range (MHz)</i>	<i>Probe Part No.</i>
200	51	7	30–53	00-968925-00
200	89	7	30–53	00-968926-01
300	51	7	46–80	00-968926-00
300	89	7	46–80	00-968926-01
400	54	7	60–105	00-968927-00
400	89	7	60–105	00-968927-01

The section has the following subsections:

- “Connecting the Probe,” [this page](#)
- “Tuning Ranges for RT MagicAngle Probes” [page 125](#)

Connecting the Probe

The following steps outline the procedure for connecting the probe, filters, and pneumatics/tachometer box (see [Figure 5](#)). Also in these instructions are steps for tuning the decoupler and observe channels when using the probe with a UNITY or VXR spectrometer. To tune the probe when used with a ^{UNITY}INOVA or UNITYplus system, refer to [Section 2.3](#), “Tuning Probes with UNITYINOVA or UNITYplus,” on page 33. These steps must be completed before running a CP/MAS experiment.

WARNING: Dangerously high rf voltage at the probe can cause serious injury or death. To avoid electrical shock, completely turn off rf and disconnect all rf cables before removing the probe.

1. If the tachometer box has an INTERNAL/EXTERNAL switch, it should be switched to the INTERNAL position.
2. Slide the upper barrel up about 2 inches (50 mm).
3. Place the rotor in the probe. Insert the probe into the magnet.
4. Connect the BEARING air supply from the pneumatics and tachometer box to the BEARING port of the probe.
5. Connect the ROTATION/DRIVE air hose to the ROTATION/DRIVE port on the probe.
6. Attach the appropriate low-pass notch filters, listed below, between the OBS connector on the probe and the PROBE connector on the preamplifier.

<i>Magnet (MHz)</i>	<i>Number of Filters</i>	<i>Filter Part No.</i>
200	2	00-948068-00
300	1	00-953049-00
400	1 or 2	00-953708-00

7. Attach the appropriate high-pass filter, listed below, to the DEC connector *on the probe*. Connect the decoupler cable from the magnet leg to the filter. Connecting the high-pass filter to the probe, instead of to the preamplifier, prevents rf interference.

<i>Magnet (MHz)</i>	<i>Filter Part No.</i>
200	00-948800-00
300	00-953374-00
400	00-953768-00

8. Connect the fiber optic cables to the pneumatics and tachometer box optical port.

9. Insert the probe into the magnet.

Varian RT CP/MAS probes have an adjustable bottom flange. The position of the flange is shown in [Figure 47](#). The collar near the top of widebore versions is for centering the probe in the magnet. The distance L, in [Figure 47](#), is from the top of the flange to the top of the probe shield.

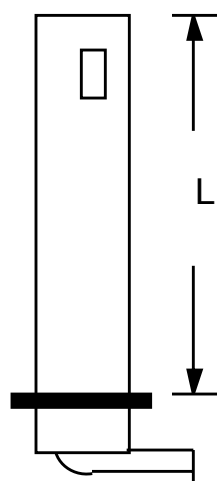
10. Start the sample spinning.

11. Tune the decoupler channel on the probe as follows:

- Enter `tn='H1' su` to set the proton frequency.
- Switch the magnet leg TUNE switch to OBSERVE and connect the probe decoupler channel to the TUNE connector on the leg.
- Adjust the red (tune) control for minimum meter deflection. (If the match is bad, the probe can be taken out of the magnet and the ^1H match capacitor, which is located along the side of the probe, adjusted.)

12. Tune the observe channel on the probe as follows:

- Change to another experiment and enter `tn='C13' su`.
- Connect the decoupler cabling back to its normal observe configuration and connect the probe observe channel to the TUNE connector on the preamplifier.



<i>Magnet (MHz)</i>	<i>Distance L</i>		<i>Distance L</i>	
	<i>Standard Bore (in.)</i>	<i>(cm)</i>	<i>Wide Bore (in.)</i>	<i>(cm)</i>
200	12.33	31.30	17.11	43.50
300	12.33	31.30	17.11	43.50
400	16.37	41.60	19.69	50.00

Figure 47. RT CP/MAS Probes Bottom Flange Position

- c. Adjust the green (tune) and blue (match) controls for minimum reflected power.
13. Repeat steps 9 and 10 (in order) until no further improvement in tuning is achievable. When finished, set the TUNE switch back to DEC and return all cables from the probe to their normal connectors.

Tuning Ranges for RT MagicAngle Probes

Capacitor and inductor sticks are provided to extend the tuning range. The nominal tuning ranges ($\pm 2\%$) are listed in [Table 28](#).

Table 28. Tuning Ranges for Varian RT CP/MAS Probes

200-MHz Probes			300-MHz Probes		400-MHz Probes	
<i>Inductor (Turns)</i>	<i>Capacitor (pF)</i>	<i>Range (MHz)</i>	<i>Capacitor (pF)</i>	<i>Range (MHz)</i>	<i>Capacitor (pF)</i>	<i>Range (MHz)</i>
none	—	49–53	none	69–82	none	88–104.7
1	10	45.6–49	9	63–71	9	80–90.0
2	18	43.0–45.6	18	58–63	18	75–81.0
3	25	41.2–43.0	28	54–58	28	70.5–75.0
4	34	39.0–41.2	37.5	51–54	37.5	68–71.0
5	100	29.9–30.8	44.5	49–51	44.5	66.0–68.0
			55.0	46.5–49	44.5	66.0–68.0
			65.0	45–46.5	64.0	62.4–64.0
			75.0	44–45	75.0	61.1–62.4

5.6 Wideline Probes

This section covers the following wideline probes for VT wideline experiments:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Tune Range (MHz)</i>	<i>Probe Part No.</i>
200	51	5	30–300	00-968568-00
200	89	5	30–300	00-968568-01
300	51	5	30–300	00-968568-00
300	89	5	30–300	00-968568-01
400	54	5	30–400	00-968568-02
400	89	5	30–400	00-968568-03

The section contains the following subsections:

- “Installing the Probe,” this page
- “Probe Tuning Hardware” page 126

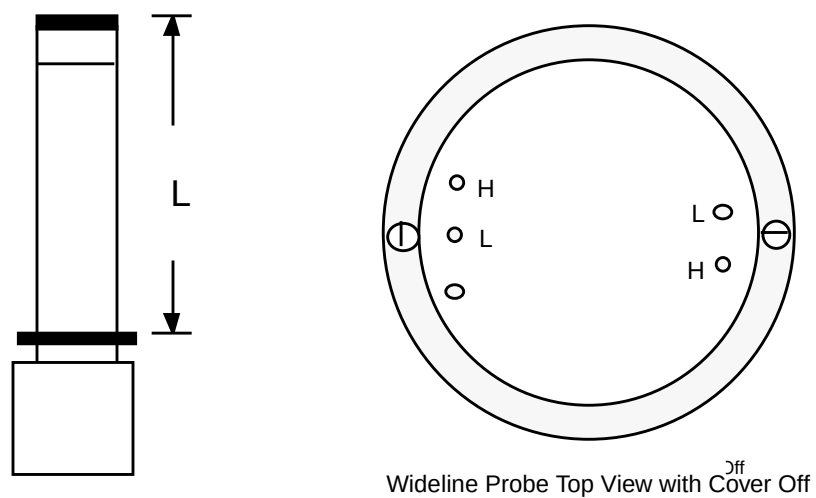
Installing the Probe

The wideline probe, shown in [Figure 48](#), is equipped for VT operation over the full temperature range of the system. Because the probe is a horizontal solenoid type, sample changing is performed by removing the probe from the magnet, removing the cap, changing the sample, and replacing the cap. The upper barrel is not used for changing samples and is left in the magnet to act as an exhaust stack for VT operation.

WARNING: Dangerous high rf voltage at the probe can cause serious injury or death. To avoid electrical shock, completely turn off rf and disconnect all rf cables before removing the probe or coils.

Probe Tuning Hardware

The standard wideline probe is a single resonance probe of the horizontal solenoid type. It accommodates samples up to 25-mm long and 5-mm in diameter. Solenoids are of a plug-in-style, allowing use of a variety of coils. Varian supplies three coils with the probe: 5-turn, 6-turn, and 10-turn. These coils can be plugged into the probe in either of two orientations (“H” or “L”), which provide two tuning ranges. Furthermore, a number of capacitor sticks are provided. These are marked with their nominal capacitance values. The combination of sticks, coil, and coil orientation allows the probe to be tuned over a frequency range that extends from the proton frequency to 30 MHz, as shown in [Table 29](#).



<i>Magnet (MHz)</i>	<i>Distance L Standard Bore</i>		<i>Distance L Wide Bore</i>	
	<i>(in.)</i>	<i>(cm)</i>	<i>(in.)</i>	<i>(cm)</i>
200	12.33	31.30	17.11	43.50
300	12.33	31.30	17.11	43.50
400	16.37	41.60	19.69	50.00

Figure 48. Wideline Probes Flange Spacing and Coil Orientation Options

Table 29. Typical Frequency Ranges, Coils, and Capacitors for Wideline Probes

<i>Frequency range (MHz)</i>	<i>Magnet (MHz)</i>	<i>Coil (turns)</i>	<i>Coil Orientation</i>	<i>Capacitor Stick (pF)</i>
130–190	400	5	L	none
105–133	400	5	L	82
260–403	400	5	H	none
273–325	200, 300	6	H	none
235–302	400	6	H	82
211–231	200, 300	6	H	82
120–207	200, 300, 400	6	L	none
98–125	200, 300, 400	6	L	11
71.7–117	200, 300, 400	10	L	none
58.1–75.2	200, 300, 400	10	L	11
56.0–66.5	200, 300, 400	10	L	18
50.5–58.5	200, 300, 400	10	L	24
44.5–50.5	200, 300, 400	10	L	33
43.5–48.2	200, 300, 400	10	L	39
40.5–44.8	200, 300, 400	10	L	43
38.5–42.1	200, 300, 400	10	L	51
38.6–41.4	200, 300, 400	10	L	56
35.4–38.2	200, 300, 400	10	L	62
35.3–37.4	200, 300, 400	10	L	68
33.1–35.3	200, 300, 400	10	L	75
31.3–33.8	200, 300, 400	10	L	82
30.1–31.7	200, 300, 400	10	L	91
29.2–30.7	200, 300, 400	10	L	100
28.9–30.0	200, 300, 400	10	L	110

5.7 Multipulse Probes for CRAMPS

This section covers the following Multipulse probes for VT (variable temperature) CRAMPS experiments:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Tune Range</i>	<i>Probe Part No.</i>
300	51	5	^1H – ^{19}F	00-993094-00
300	89	5	^1H – ^{19}F	00-993094-01
400	54	5	^1H – ^{19}F	00-992618-00
400	89	5	^1H – ^{19}F	00-992618-00

Multipulse probes are standard CRAMPS probes manufactured by Varian. These probes have a VT range from -120° to $+160^\circ$ C and can typically tune from the ^{19}F frequency to that of ^1H . The probe is optimized for ^1H and is essentially background free for that nucleus. In order to achieve low ^1H background, many components are made of a fluorocarbon, so that a high ^{19}F background exists. This may not necessarily preclude ^{19}F line narrowing experiments, but care must be taken.

WARNING: Dangerous high rf voltage at the probe can cause serious injury or death. To avoid electrical shock, completely turn off rf and disconnect all rf cables before removing the probe.

WARNING: Spinner rotors have high kinetic energy and can disintegrate at any time. To prevent possible cuts or injuries to eyes (including possible blindness), wear a certified full-faced safety shield and cover all exposed skin while observing a rotating sample

Multipulse probes have a 5-mm rotor and are designed to spin to 8 kHz. In order to have a fast recovery, the Q is of the order of 10–30, controlled by a Q damping box connected to the PROBE port. As a result, the probes do not have high sensitivity. This is not normally an issue in multipulse experiments, where proton is an abundant spin. Where rare spin conditions apply, the damping box can be removed, in which case the ringdown time is longer.

Preparing the Sample

Final tests are performed using a solid sample of adipic acid. Sample preparation is crucial to obtaining the resolution specification. The sample, a white powder, must be packed centrally in the rotor and be only 1 mm long; this is done as shown in the following steps.

1. Pack either a spacer or some inert material *with no protons* to the center of the rotor. Tamp down this sample carefully.
2. Add a small amount of adipic acid and tamp the acid down.
3. Fill the rotor with more inert material and insert the cap.
4. Replace the benzene sample with the adipic acid sample. *Do not retune the probe.* Put the probe in the magnet and spin the sample at 1550 Hz.
5. Run the “mrev8 Calibration” procedure as described in the *Solids Modules Installation* manual that shipped with your system.
6. Set `tof` to the best value as determined in the `mrev8` procedure and set the following parameters: `np=512 tau=3.5 fn=4096 nt=1`

7. Acquire a spectrum. Note that the `dScale` values are wrong! Vary `tof` to get the best resolution of the right-hand peaks.
8. Place a cursor on each peak of the doublet and enter `scalesw=sfrq/delta` to set the scale factor.
9. Place the cursor on the right-most peak and enter `rl(2p)` to reference it. The spectrum should now have resonances at 2 ppm, 3 ppm, and about 14 ppm.
10. Save the FID for later reference, using `svf(file)`, where `file` is the name you select for file to hold the FID; for example, `svf('testdata1')`.

5.8 Doty VT CP/MAS Probes

This section covers the following Doty probes for VT CP/MAS probes:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Tune Range (MHz)</i>	<i>Probe Order No.</i>
200	51	7	6.3–81 (^{103}Rh – ^{31}P)	00-957828-00
200	89	7	6.3–81 (^{103}Rh – ^{31}P)	00-957828-01
300	51	7	9.4–122 (^{103}Rh – ^{31}P)	00-957829-00
300	89	7	9.4–122 (^{103}Rh – ^{31}P)	00-957829-01
400	54	5	12.6–162 (^{103}Rh – ^{31}P)	00-957831-00
400	89	5	12.6–162 (^{103}Rh – ^{31}P)	00-957831-01

Note: The Doty probe manual supersedes any description of mechanical operations in this manual.

Connecting the Probe

The following steps outline the procedure for connecting the probe, filters, and pneumatics/tachometer box (see [Figure 6](#)). Also in these instructions are steps for tuning the decoupler and observe channels when using the probe with a UNITY or VXR spectrometer.

The Doty Scientific CP/MAS probe for the 200-, 300-, and 400-MHz magnet requires gas supply lines for BEARING and ROTATION/DRIVE connectors.

To tune the probe when using it with a UNITYINOVA or UNITYplus system, refer to [Section 2.3, “Tuning Probes with UNITYINOVA or UNITYplus,”](#) on page 33.

Follow these steps before running a CP/MAS experiment.

1. If the pneumatics/tachometer box has an INTERNAL/EXTERNAL switch, switch it to EXTERNAL and connect the Doty tachometer amplifier to the BNC connector.
2. Slide the upper barrel up about 50 mm (2 in.).
3. Place the rotor in the probe. Insert the probe into the magnet.
4. Connect the BEARING air supply from the pneumatics and tachometer box to the BEARING port of the probe.
5. Connect the ROTATION/DRIVE air hose to the ROTATION/DRIVE port on the probe.
6. Attach the appropriate low-pass notch filters, listed below, between the OBS connector on the probe and the PROBE connector on the preamplifier.

<i>Magnet (MHz)</i>	<i>Number of Filters</i>	<i>Filter Part No.</i>
200	2	00-948068-00
300	1	00-953049-00
400	1 or 2	00-953708-00

7. Attach the appropriate high-pass filter, listed below, to the DEC connector *on the probe*. Connect the decoupler cable from the magnet leg to the filter. Connecting the high-pass filter to the probe, instead of the preamplifier, prevents rf interference.

<i>Magnet (MHz)</i>	<i>Filter Part No.</i>
200	00-948800-00
300	00-953374-00
400	00-953768-00

8. Connect a cable from the SPIN DETECTION port on the probe to the Doty preamp. Then connect the OPTIC output of the preamp to the DOTY IN connector on the tachometer box.
9. Insert the probe into the magnet and start the sample spinning (see [Section 5.2, "Sample Spinning,"](#) on page 109).
10. Tune the decoupler channel on the probe as follows:
 - a. Enter `tn= 'H1' su` to set the proton frequency.
 - b. Switch the magnet leg TUNE switch to OBSERVE and connect the probe decoupler channel to the TUNE connector on the leg.
 - c. Adjust the blue (tune) and the green (match) controls for minimum meter deflection.
11. Tune the observe channel on the probe as follows:
 - a. Change to another experiment and enter `tn= 'C13' su`
 - b. Connect the decoupler cabling back to its normal observe configuration and connect the probe observe channel to the TUNE connector on the preamplifier.
 - c. Adjust the silver (tune) and the red (match) controls for minimum meter deflection.
12. Repeat steps 10 and 11 (in order) until no further improvement in tuning is achievable. When finished, set the TUNE switch back to DEC and return all cables from the probe to their normal connectors.

Tuning Ranges

The color coding and position of the various adjustment rods on Doty probe also differ from those of Varian probes. Because these components of the probe change from time to time, be sure to refer to the manual shipped with the Doty probe.

Chapter 6. Probes for Microimaging NMR

Sections in this chapter:

- 6.1 “Installing the Gradient Coils” [this page](#)
- 6.2 “Inserting the Sample” [page 134](#)
- 6.3 “Installing the Probe” [page 135](#)
- 6.4 “Tuning the Probe” [page 135](#)

This chapter describes installation, cable connections, and sample insertion for the following Varian proton probes for microimaging NMR:

<i>Magnet (MHz)</i>	<i>Bore (mm)</i>	<i>Sample Size (mm)</i>	<i>Sample Type</i>	<i>Probe Part No.</i>
300	89	25	solid	00-990700-00
300	89	25	liquid	00-990700-00
400	89	25	solid	00-991014-00
400	89	25	liquid	00-991014-00

6.1 Installing the Gradient Coils

[Figure 49](#) illustrates the probe and gradient coil configuration.

WARNING: When installing the gradient coils, make sure the gradient amplifiers are either switched to off or standby. The gradient amplifiers produce potentially lethal amounts of power.

1. Remove the upper barrel (it is not used in microimaging).
2. Insert the gradient coil unit into the room temperature (RT) shim coils.
Make sure the indicated X direction of the gradient coils lines up with the X direction of the magnet.
3. Tighten the mounting flange thumbscrews to the end flange of the RT shim coils—be sure to tighten the thumbscrews equally.
4. Open the junction unit and connect J806 (inside the box) and the 15-pin D shell J802 (mounted to the outside of the box).
5. Connect an air hose to the tee between S801 and the pressure gauge (see [Figure 49](#)). The gauge should read between 10 and 20 psi; switch S802 needs more than 10 psi to close.

6.2 Inserting the Sample

To insert the sample in a microimaging probe:

1. Slide the spacer on the probe.
2. Gauge the sample with the sample gauge as follows:
 - a. Put the gauge ring on the sample tube.
 - b. Insert the sample tube into the sample gauge.
 - c. Position the desired sample location with the rf coil center line (marked on the gauge and shown in [Figure 50](#)).
 - d. Hold the sample tube with the gauge and move the ring until it touches the upper end of gauge.
3. Insert the sample into the probe and make sure that the sample gauge ring rests against the upper insulator in the probe. Note that the sample can also be loaded

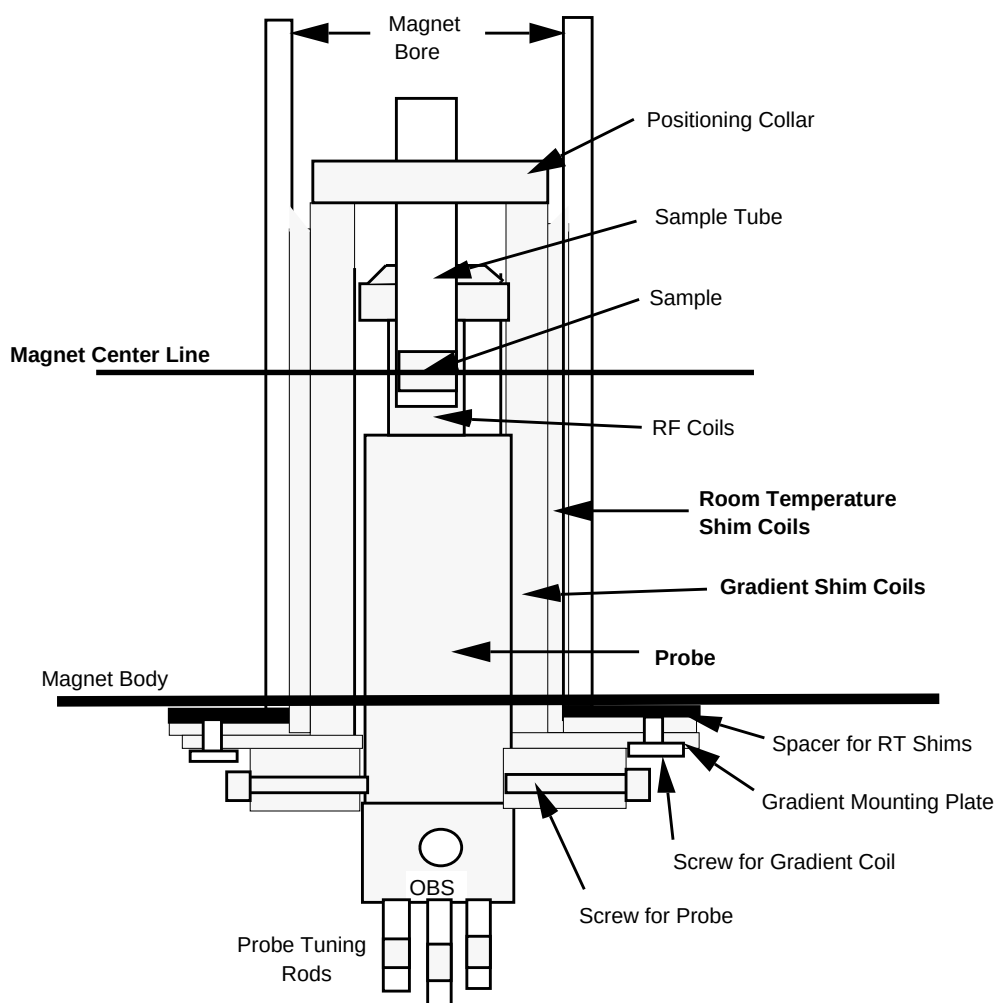


Figure 49. Microimaging Probe and Gradient System

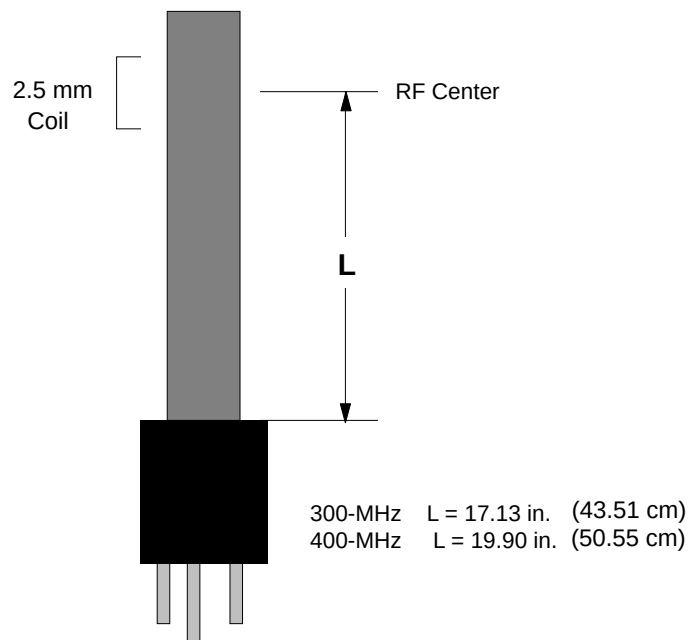


Figure 50. RF Coil Center of Microimaging Probe

manually by reaching down through the top of the magnet bore after the probe is installed.

6.3 Installing the Probe

To install the microimaging probe:

1. Insert the probe into the bottom of the gradient coil unit. Push the probe upward until the spacer touches the lower flange of the gradient coil unit. Secure the probe with the thumbscrews.
2. Connect the rf input to the OBSERVE connector on the probe (see [Figure 51](#)).
3. Connect the VT cable from the VT controller to the 9-pin connector on the probe.
4. Connect the VT nitrogen gas hose to the stub tee on the glass dewar extending down from the bottom of the probe.
5. Connect the body-cooling air hose to the hose barb extending downward from the base of the probe.
6. Close the junction unit. Make sure the cover does not pinch the air tubing.

6.4 Tuning the Probe

The microimaging probe has an indicator base as shown in [Figure 51](#). This probe has three tuning rods extending from the bottom of the probe, two of these are “tune” rods, the other is a “match” rod. All three rods are tuned alternately until the reflected power on the leg meter arrives at a minimum when the probe is connected in the TUNE mode.

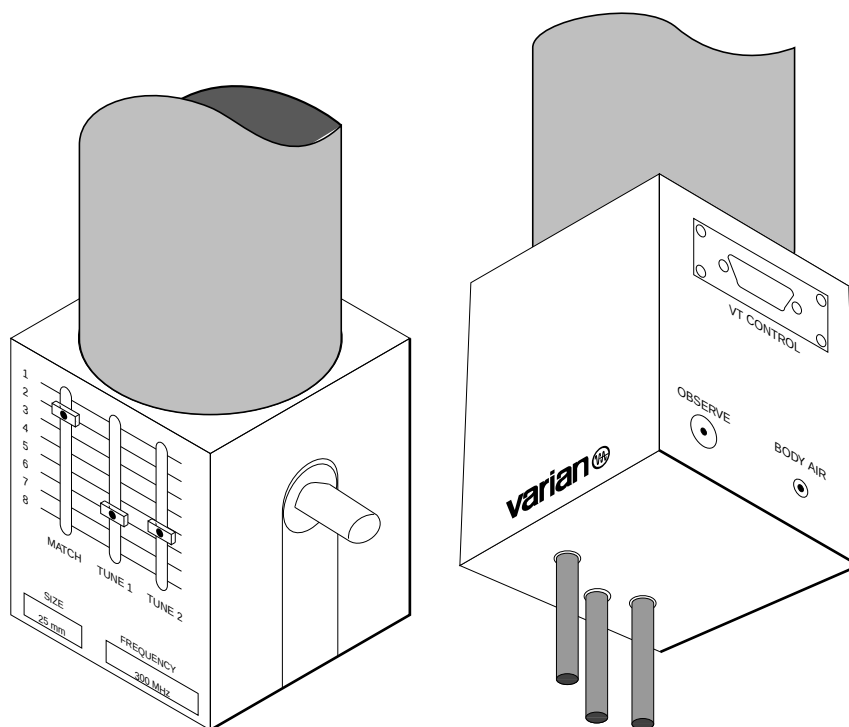


Figure 51. Microimaging Probe Tuning Controls and Connectors

Tuning further requires adjusting the TUNE 1 and TUNE 2 capacitors so that they show the same number (to within one division) on the indicators. Each rod moves an indicator on a scale on the probe front.

After the probe has been tuned, write down the indicator positions by recording the values. You can return to the approximate tuning for each type of sample in the future by resetting the rods to the numbers you wrote down.

For probe tuning, refer to the appropriate section for your system:

- UNITYINOVA, UNITYplus – See [Section 2.3, “Tuning Probes with UNITYINOVA or UNITYplus,”](#) on page 33.
- UNITY, VXR – See [Section 2.5, “Tuning Probes with UNITY or VXR,”](#) on page 40.

Chapter 7. Maintenance and Troubleshooting

Sections in this chapter:

- 7.1 “Probe Maintenance” [this page](#)
- 7.2 “Probe Troubleshooting” [this page](#)
- 7.3 “CP/MAS Solids Probe Troubleshooting” [page 139](#)

This chapter describes general maintenance and troubleshooting for probes.

7.1 Probe Maintenance

The following maintenance is recommended for probes:

- Periodically check the probe and upper barrel for proper alignment and mating.
- If, during normal operation, the sample spin rate changes dramatically or a sample does not eject, check steps 1 through 5 of the [Section 1.3, “Installing the Probe and Upper Barrel,”](#) on page 18.

CAUTION: Do not use any kind of tool or solvent to clean the probe because this may cause damage to sensitive parts.

- Clean the probe periodically by carefully inverting the probe and spraying the insert area with isopropanol (only), drain, and allow to air dry. If this procedure does not sufficiently remove contaminants, consult Varian Field Service for assistance.

7.2 Probe Troubleshooting

This section describes potential system problems and how to fix them.

Leaks in Tubing

Possible causes and solutions:

- Be sure that the tubing is pushed all the way in. This is often difficult to do with two fingers, especially when the system is pressurized. Pressure tends to push the tubing away from the fitting. To repair a leak, use a pair of pliers to grasp the tubing gently and push it toward the fitting (remember that you are working within a strong magnetic field, and it is possible for the pliers to be pulled from your hand).
- When a piece of tubing is pushed in and pulled out several times, the tip of the tubing may wear out. If this happens, cut about 6 to 12 mm (1/4 to 1/2 in.) off the tubing.

When cutting the tubing, make sure the cutting surface is flat and square in order to assure a tight seal.

Sample Does Not Spin

Possible causes and solutions:

- Bearing or system air pressure is too low.
- Upper barrel is not mating properly or is misaligned with the probe or shim set.
- Sample tube length is excessive.
- Spinner bearing surface is dirty. Remove the upper barrel to inspect. Clean the bearing surface with a cotton swab and isopropanol solvent.
- Dirty or non-precision sample tube: clean or replace.
- Dirty or damaged turbine: clean or replace.

Sample Does Not Eject

Possible causes and solution:

- Air leak somewhere in system, or low air pressure.
- VT or cooling air not connected.
- Loose or pinched hose on the upper barrel. Remove upper barrel and inspect all hose connections.
- Upper barrel not mating with probe. Remove upper barrel and probe and clean.
- Broken sample. Remove upper barrel and probe. Clean both the upper barrel and the probe. If any glass is in the probe, contact Varian field service for repair.

Note that if the sample cannot be ejected and the upper barrel has to be removed with the sample in place, use care not to break off the sample. The sample tube will extend below the end of the upper barrel.

Sample Spins But Does Not Regulate (White Turbines Only)

The spinner should regulate at most speeds between 15 and 50 Hz under normal conditions.

Possible causes and solutions:

- Sample tube is rubbing on the probe insert. To correct this, follow this procedure.
 - a. Enter the command *acqi* and go to the lock display.
 - b. Set the spinner speed to some value above 50 Hz. If the spinner speed reads 80 Hz, skip to step 5.
 - c. While pushing down on the sample delivery barrel, rotate it about 1/10 turn.
 - If the spinning speed increases beyond 50 Hz, skip to step 5.
 - If the spinner speed increases but remains below 50 Hz, repeat step 3, rotating the upper barrel another 1/10 turn.
 - If the spinner speed decreases, then repeat step 3, but rotate the barrel in the opposite direction.
 - d. If the spinner won't reach speed using the method of step 3, try shifting the upper barrel mounting plate (collar) slightly and repeat step 3.

- e. If the spinner rotates at 50 Hz or faster, it should regulate properly and can now be used for experiments. The sample tube, however, may still be rubbing against the insert. Follow step 6 to check for rubbing.
- f. As the sample spins at 50 Hz or faster, rapidly turn the spinner speed down to some value less than 10 Hz, without letting the requested speed drop to zero. Observe the resulting spinner speed decay rate. The speed of a sample that is not rubbing should not decay by much more than 5 Hz with each display update under 45 Hz. If the spinning rate decays by 15 Hz or more with each update, the method of step 3 can be used to “fine tune” the alignment of the upper barrel.
- Spinner is vibrating. Spinner resonant frequencies generally range from 30 to 45 Hz. If the spinner will not regulate at a certain speed within this range, try spinning at some other speed.
- Bearing or system air pressure is not adjusted properly.

7.3 CP/MAS Solids Probe Troubleshooting

WARNING: Spinner rotors have high kinetic energy and can disintegrate at any time. To prevent possible cuts or injuries to eyes (including possible blindness), wear a full-face certified safety shield and cover all exposed skin while observing spinning rotors.

Sample Does Not Spin Up to 4 kHz

- Try increasing bearing pressure, checking that the cap is on tight, or repacking the sample.

γB_2 Too Low

- Carefully increase `level2`; However, you can damage the 100 W amplifier if you increase `level2` too far. Bear in mind that an increase of 3 is doubling the power.

Pulse Width Too Long

- If `xpol='n'`, check the probe tuning and the output power of the amplifier at the probe, and then increase `tpwr`.
- If `xpol='y'`, decrease spinning speed to 2000 Hz and determine the Hartmann-Hahn condition.

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